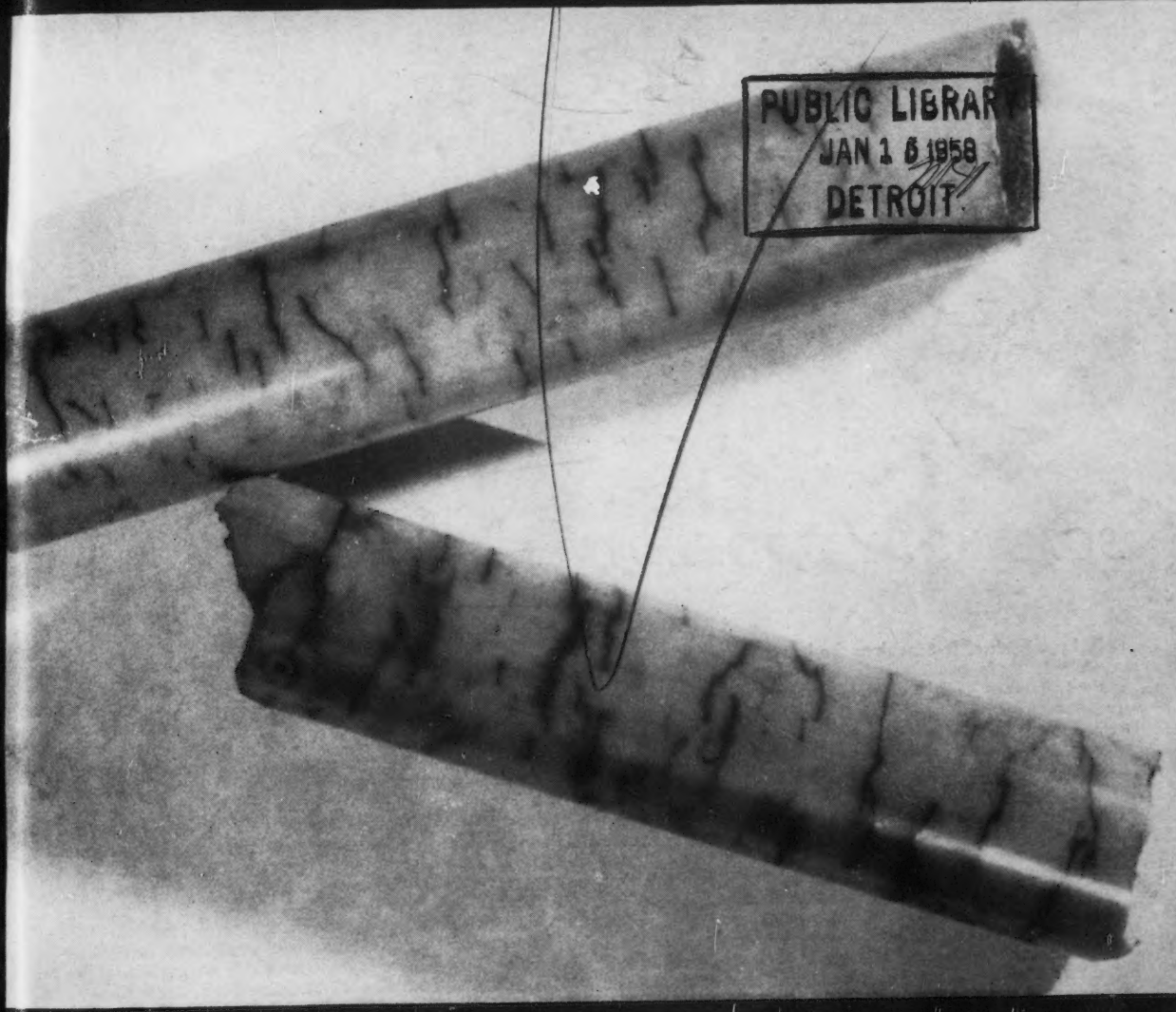
Corrosion

OFFICIAL PUBLICATION OF
NATIONAL ASSOCIATION OF CORROSION ENGINEERS



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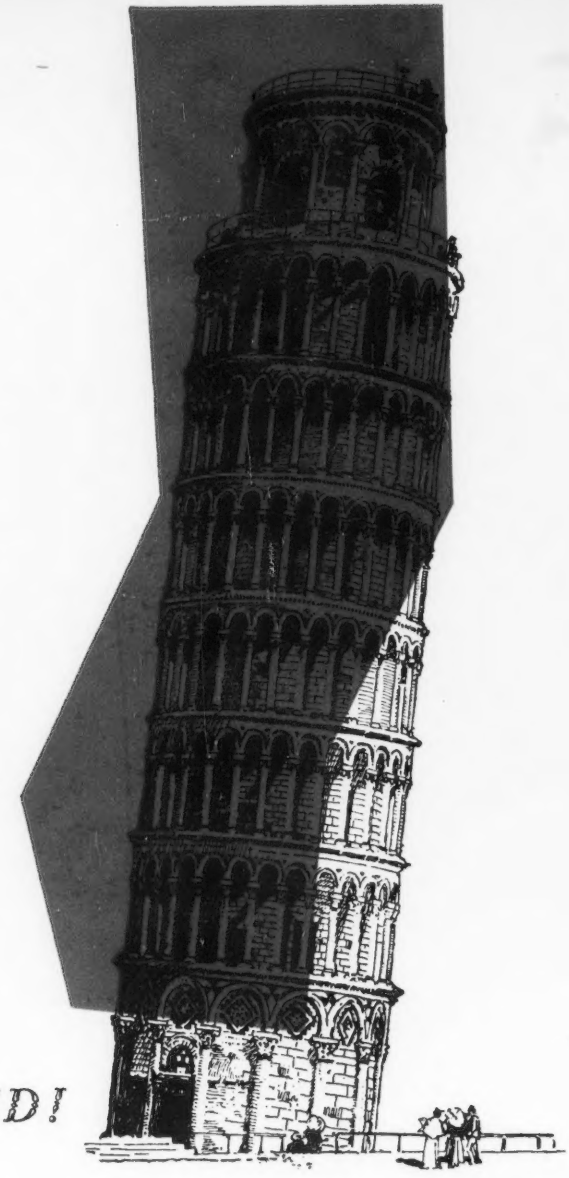

SOMEBODY GOOFED!

Only by a freak of fortune does the leaning tower of Pisa still stand. A faulty foundation was not apparent until the structure was three stories high. It is believed the architects were then forced to compensate by adding weight to the opposite side to save the building.

Are you building corrosion problems into your plant structures and equipment that will inevitably have to be compensated for by tremendous replacement costs? Many of these costs can be eliminated *at the planning stage* by taking advantage of the services of an expert in the field of corrosion resistant coatings... the Amercoat Sales Engineer.

The Amercoat Sales Engineer is trained in the principle that in corrosion engineering, too, foundations are all-important. You can put this factory-trained man on your staff, so to speak, without cost or obligation. His advice will be truly objective since it is based on a knowledge of all types of protective coatings and more than 43,000 case histories in our files. You can be sure that the Amercoat coating he recommends, whether vinyl, phenolic, epoxy or any other type, will be the right one for *your* job, and the most economical on the basis of cost per square foot per year.

Therefore, to effect important economies in specifying protective coatings you are cordially invited to call on your Amercoat Sales Engineer... *right at the beginning.* He will analyze all pertinent data concerning your corrosion problems based on our nearly 20 years of corrosion control experience, and give you a comprehensive recommendation. In addition to recommending the proper coatings, he will assist you in writing complete specifications. However, this is not the full extent of his service: he will also be available for consultation at the job site to insure proper application.

There are more than 70 Amercoat Sales Engineers and Distributors located throughout the country. It will pay you to talk to one of these men whether you are building a new plant or are interested in the efficient maintenance of existing facilities. The same service is available and equally important savings can be realized in both cases.

May we send you the name of the Amercoat man nearest you? Literature containing many helpful suggestions for the design and corrosion engineer is also available on request. Write to: Amercoat Corporation, 4809 Firestone Blvd., South Gate, Calif.

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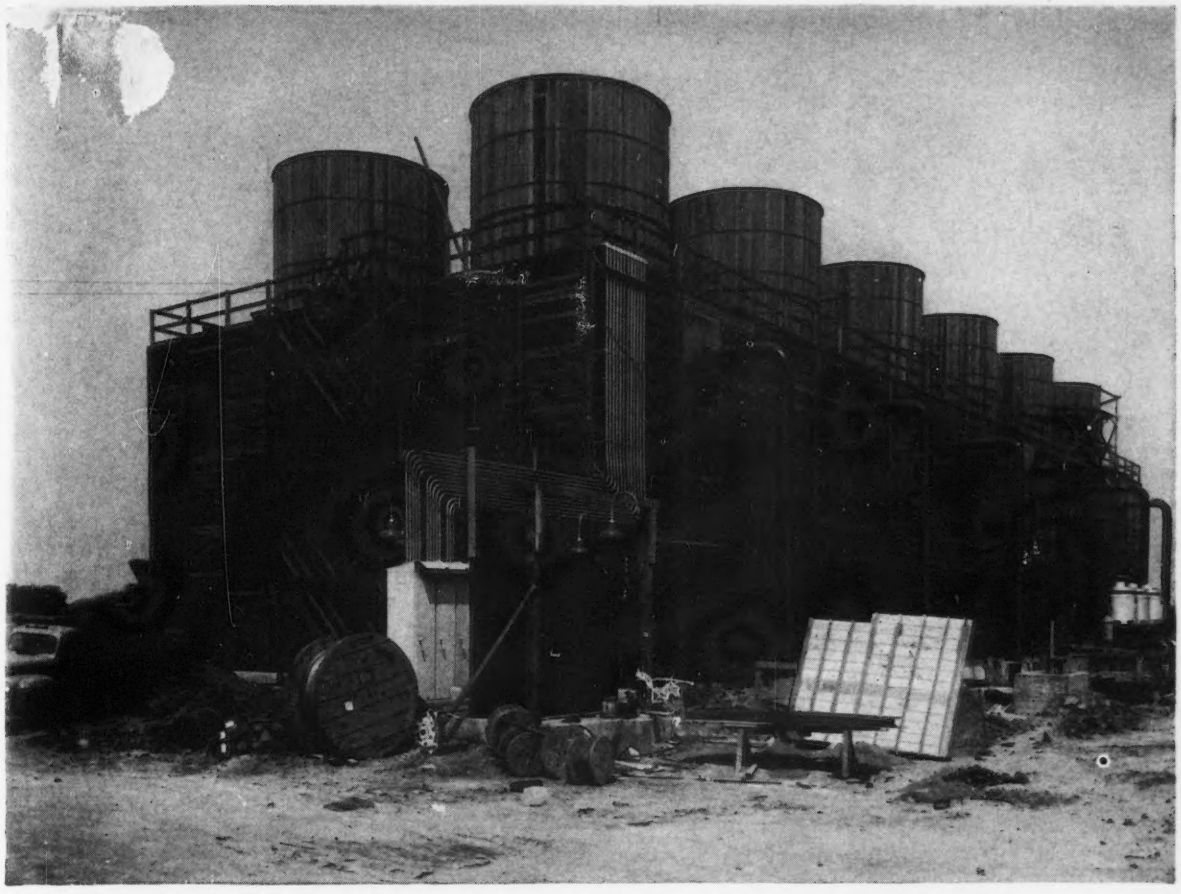

The time to stop corrosion is before it starts

Long before the first drop of water contacts metal in any large recirculating water system, steps should be taken to prevent corrosion.

Early in the design stage, plan on adding chromate inhibitors to recirculating water—from the moment operation starts. That way, clean metal stays clean. Delay, and the metal gets blanketed by rust and scale, which makes it difficult for any inhibitor to reach and protect the metal.

Chromates work two ways: they make waters non-corrosive while making metal surfaces corrosion-resistant. Chromates are inexpensive to use—they do their work at maintained concentrations of 100 to 400 parts per million. These inhibitors may be introduced either as sodium chromate or bichromate.

For literature on Mutual chromates in recirculating water systems, or in other applications, mail the coupon below.

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"Those Galvomag anodes give us the added punch we need for our high-resistivity soil"

If you've got an underground corrosion problem, Galvomag® magnesium anodes are almost always the best solution.

Galvomag, the Dow high-potential anode that delivers 25% more current than conventional anodes, gives continuous protection to underground equipment—and it's easy on your maintenance budget. These more powerful anodes also provide extra punch and throwing power in high-resistivity soils. In normal soils they insure against corrosion with fewer anode units. Either way, you save on your original investment for cathodic protection.

Contact one of the Dow magnesium anode distributors listed below for detailed information and technical assistance, or write directly to us. THE DOW CHEMICAL COMPANY, Midland, Michigan, Department MA-1428GG-4

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devoted entirely to corrosion research and control

Published monthly as its official journal, by the National Association of Corrosion Engineers, Inc., at Houston, Texas, U. S. A., as a permanent record of progress in corrosion control.

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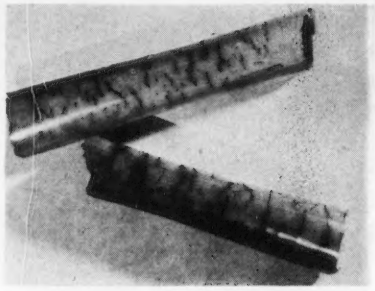

THIS MONTH'S COVER—Two Sections of 2¼-inch OD, ¼-inch wall extruded, linear high molecular weight polyethylene pipe stress cracked after 149 days' exposure to wet chlorine gas and saturated NaCl brine splash at approximately 100 C. Cracks are indicated by the dye penetrant method. Photograph from Hooker Electrochemical Co., Niagara Falls, N.Y.

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Many and great are the claims that are being made for "high potential" magnesium anodes. Yet it is a proved fact that they are economically unsound in more than 90% of the cases where magnesium anodes are required.

There are three reasons for this:

1. Magnesium anodes are essentially packaged electrical energy and their efficiency is measured by the amount of that electrical energy which is recoverable as useful current. The advantages of an anode with high effjciency are obvious. Under all conditions, the most efficient anode will provide either a longer life at a given current flow, or a larger current flow at a given anode life. High potential magnesium anodes produce an average current efficiency of 48.59%, whereas, H-1 alloy anodes produce a minimum current efficiency of 60%. In other words, H-1 alloy anodes deliver more ampere hours of protection per pound of metal consumed. When attaching a dollar and cents value to anode efficiency, it is easily understood that greater protection and economy is obtained through the use of H-1 alloy anodes.

2. It is current delivered, not the potential, that stops corrosion. Potential is necessary only to overcome resistance and in order to deliver current to the structure to be protected. Logically then, reducing the resistance reduces the requirement for potential. This resistance decreases rapidly by extending the length of the anode. Therefore, with 50% increased length, with no increase in anode weight, a higher current output can be obtained from the H-1 alloy anode than from the "High Potential" anode.

3. Most corrosion is caused by galvanic cells occurring in low resistance soils and in sea water. High potential anodes generate 25% more immediate current punch than is necessary to protect against corrosion in these cases. The fact that H-1 alloy anodes deliver a proper current level to protect against corrosion in low resistance soils and in sea water has been proved over many years in thousands of installations.

These three reasons mean that in 90% of all magnesium anode installations the H-1 alloy anode has a life expectancy of 10 years as opposed to a maximum 5.5 year life expectancy of the high potential anodes.

It also means that the H-1 alloy anode has a greater current efficiency . . . that it delivers more useful ampere hours of protection per pound of metal consumed.

In low resistivity soils and in sea water where most corrosion occurs — high potential anodes develop more immediate current than is actually needed to obtain full protection.

It is a recognized, undisputed fact that the greatest amount of stored useful electrical energy is obtained from H-1 alloy anodes. It is this more useful current that offers the protection that is needed.

Study your anode purchases. Are you actually paying more for protection you do not need? In many cases you will find that the installation of high potential anodes is costing you more money and offering less protection than the H-1 magnesium anode.

Don't be misled into thinking that a slightly greater potential means more stored electrical energy.

We can prove to you that Standard Magnesium's Vibra-Pak H-1 alloy anode will give you more useful electrical energy — with resulting longer life, more protection and greater economy.

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T-2G Coal Tar Coatings for Underground Use
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T-2H Asphalt Type Pipe Coatings
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P. E. Reynolds, Vice Chairman; Pacific Gas & Electric Co., 245 Market St., San Francisco, Cal.

T-2H-1 Methods to Classify Environmental Conditions
(See chairman T-2H)

T-2H-2 Criteria for Reporting on and Evaluating Underground Coatings
(See chairman T-2H)

T-2H-3 Specifications
(See chairman T-2H)

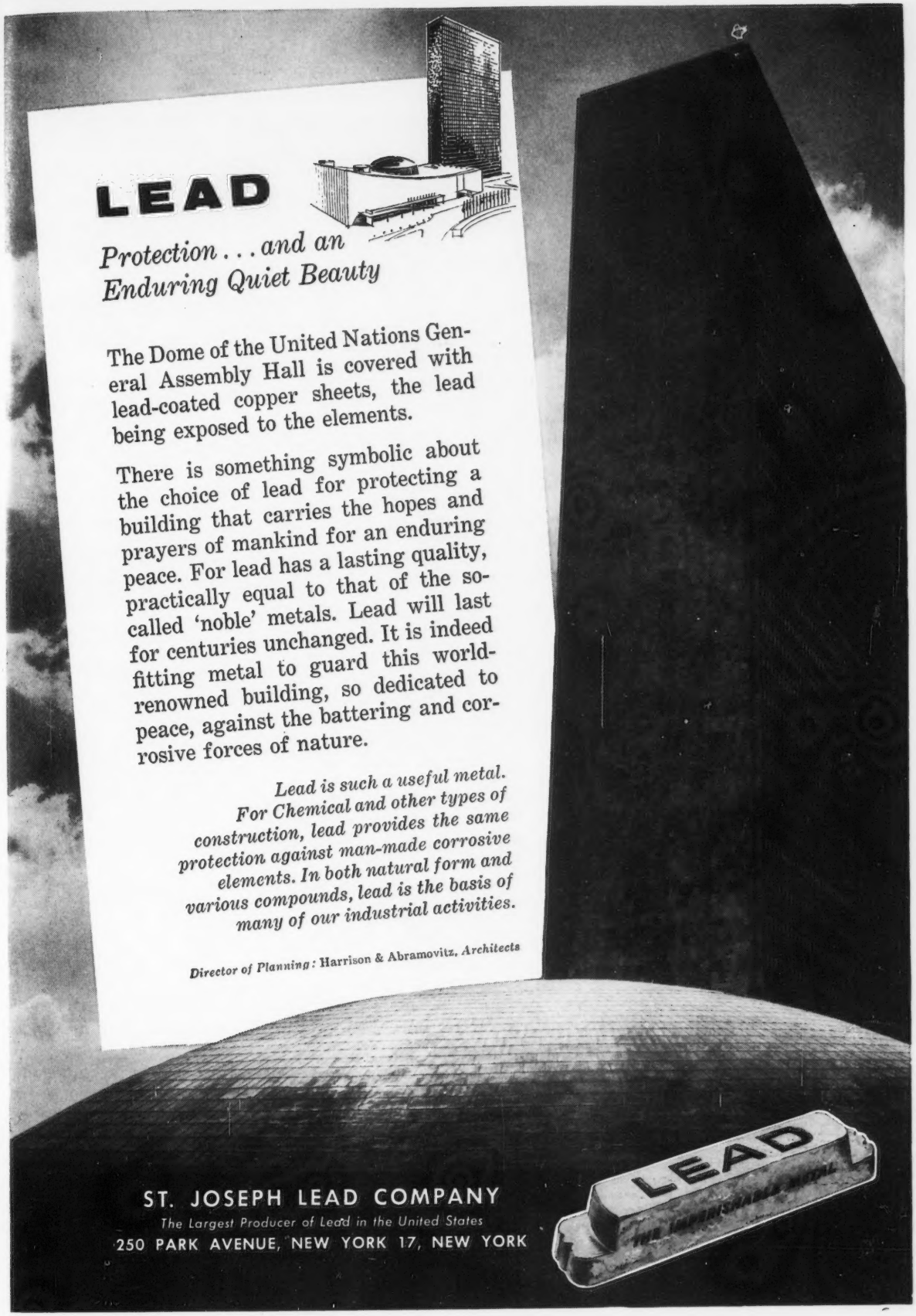
T-2H-4 Internal Pipe Treatment Specifications
F. B. Burns, Chairman; Kerr-McGee Oil Industries, Inc., Box 125, Wynnewood, Okla.

T-2J Wrappers for Underground Pipe Line Coating
Clark A. Bailey, Chairman; Johns-Manville Sales Corp., 22 East 40th St., New York 16, N. Y.
O. W. Wade, Vice Chairman; Transcontinental Gas Pipe Line Corp., Box 296, Houston, Texas

T-2J-1 Asbestos Felt
P. A. Meyer, Chairman; Nicolet Industries, Inc., 70 Pine St., New York, N. Y.

T-2J-2 Glass Wrap
E. J. Pennell, Chairman; Owens-Corning Fiberglas Corp., 902 American Airlines Bldg., Tulsa, Okla.

(Continued on Page 8)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T-2J-3 Glass Base Outer Wrap
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T-2J-4 Rag Felt
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T-2J-5 Rock Shield
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T-2K-2 Research and Development
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T-2K-3 History and Results
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T-3A-2 Methods of Testing & Screening Corrosion Inhibitors
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T-3A-3 Materials Available for and Application of Corrosion Inhibitors
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T-3B Corrosion Products
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T-3C Annual Losses Due to Corrosion
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T-3D Instruments for Measuring Corrosion
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T-3E Railroads
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T-3E-2 Corrosion in Railroad Hopper Cars
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T-3F Corrosion by High Purity Water
R. U. Blaser, Chairman; Research Center, Babcock & Wilcox Co., Box 835, Alliance, Ohio.

T-3F-1 Facilities for Production of High Purity Water

T-3F-2 Inhibitors
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T-3F-3 Corrosion Products
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T-3F-4 General Corrosion Problems
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T-3F-5 Declassification
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T-3F-6 Intercommittee Activities
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T-3G Cathodic Protection
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T-3G-1 Cathodic Protection of Hull Bottoms of Ships
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T-3G-2 Cathodic Protection of Heat Exchangers
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T-3G-3 Cathodic Protection of Process Equipment
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T-4A Effects of Electrical Grounding on Corrosion
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T-4A-1 Recommendations for Materials to Be Used for Electrical Grounding to Reduce Corrosion
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T-4A-2 Recommendations for Grounding on Private Premises
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T-4B Corrosion of Cable Sheaths
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T-5B-3 Oil Ash Corrosion
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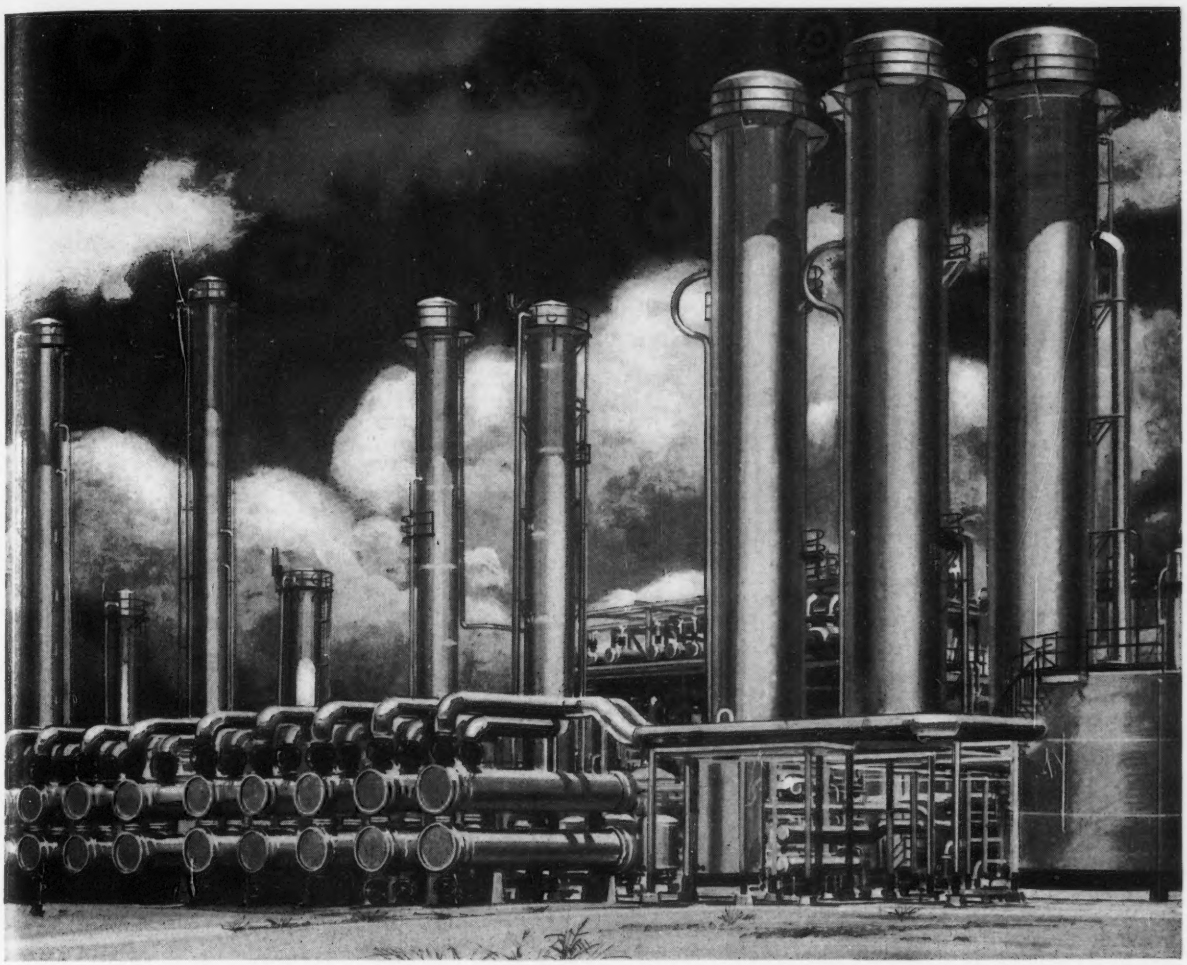

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T-5B-5 Corrosion by Molten Salts and Metals

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T-5C Corrosion by Cooling Waters

T-5C-1 Corrosion by Cooling Waters (South Central Region)

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T-5D-6 Organic Chemicals

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T-6A-3 Vinylidene Chloride Polymers

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T-6A-4 Phenolics

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T-6A-8 Methyacrylates

T-6A-9 Furanes

T-6A-10 Polyesters

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T-6A-13 Chlorinated Rubbers

Appointment Pending

T-6A-14 Organic-Brick Covered

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T-6A-15 Rigid Vinyls

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T-6A-16 Coal Tar Coatings

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T-6A-17 Polyurethanes

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T-6A-18 Hypalon

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T-6B Protective Coatings for Resistance to Atmospheric Corrosion

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T-6B-1 Linseed and Other Drying Oils

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T-6B-3 Straight Phenolic Oil Varnish

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T-6B-4 Modified Phenolic Oil Varnish

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T-6B-6 Modified Alkyd Varnish

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T-6B-7 Epoxy Esters

T-6B-8 Epoxy (Amine Cured)

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T-6B-9 Chlorinated Rubber

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T-6B-11 Metallic Silicates

T-6B-12 Coal Tar

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T-6B-14 New Developments

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T-6C Protective Coatings for Resistance to Marine Corrosion

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T-6D-1 Economics of Maintenance Painting

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T-6D-3 Paint Programs

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T-6F-3 Inspection

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T-6F-4 Safety

(Chairman to be announced)

T-6F-5 Application

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T-6G Surface Preparation for Organic Coatings

Joseph Bigos, Temporary Chairman; United States Steel Corp., Applied Research Lab., Monroeville, Pa.

T-6H Glass Linings and Vitreous Enamels

T-6J Protective Coating Application Problems

T-6J Los Angeles Area, Protective Coating Application Problems

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T-6J-2 Application Procedure

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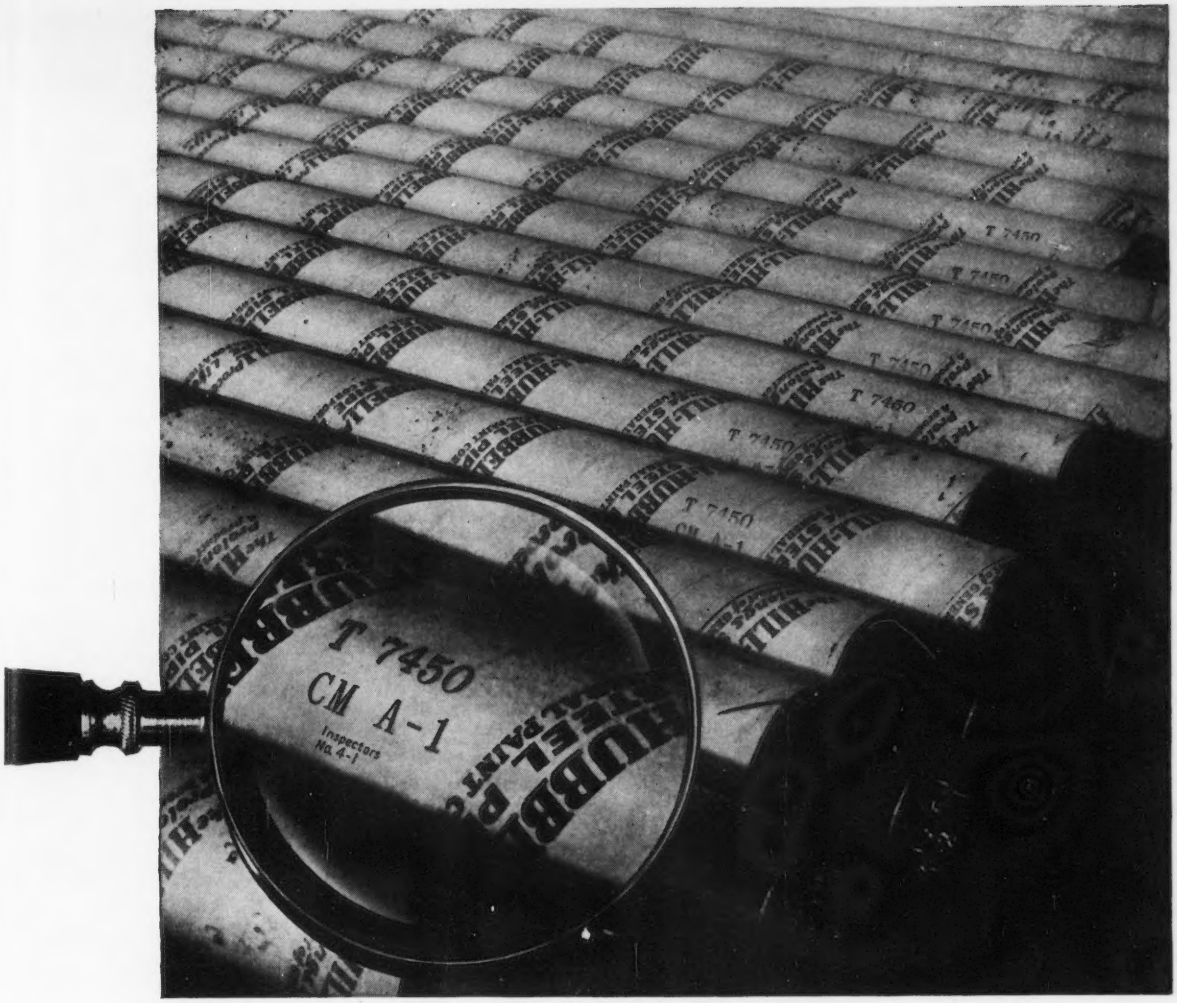

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T-6J-4 Cost Evaluation

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T-6K Corrosion Resistant Construction With Masonry and Allied Materials

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T-9B Preservations and Their Appraisal

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TECHNICAL ARTICLES SCHEDULED FOR PUBLICATION IN FEBRUARY

Nitrite Inhibition of Corrosion: Some Practical Cases by T. P. Hoar

Effect of Inhibitors in Fuming Nitric Acid on Corrosion and Oxidation by Tin Boo Yee

Study of the Compatibility of Floating-Type Inhibitors and Cathodic Protection by E. R. Streed

Variables Influencing Corrosivity of Oil and Gas Wells by H. E. Waldrip and J. A. Rowe

Staining of Copper and Brass by Einar Mattson

Protective Linings for Steel Shipping Containers by L. J. Nowacki

Corrosion of Stainless Steel and Its Suppression in Ferric Salts by Michael A. Streicher

Electrochemical Deterioration of Graphite and High Silica Iron Anodes in Sodium Chloride Electrolytes by S. Tudor, W. L. Miller, A. Ticker and H. S. Preiser

Corrosion Products of Iron in Hydrogen Sulfide Environments by F. H. Meyer, R. L. McGlasson, O. L. Riggs and J. D. Sudbury

Iron-Silicate Slag Network Gives Wrought Iron Corrosion Resistance by E. P. Best

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Flowing	West Texas	420	—	—	Twice a week	.13¢
Gas	East Texas	25	Trace	22.9	Twice a week	4.6¢*
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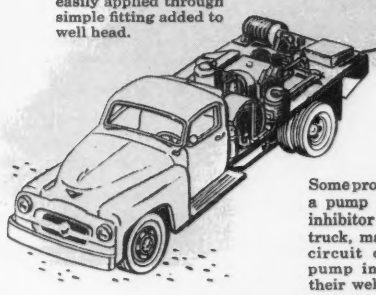

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HYDROGEN ABSORPTION, EMBRITTLEMENT and FRACTURE of STEEL

By Arnold E. Schuetz, Research Department, Bridgeport Brass Company, Bridgeport, Conn. and W. D. Robertson, Hammond Metallurgical Laboratory, Yale University, New Haven, Conn.

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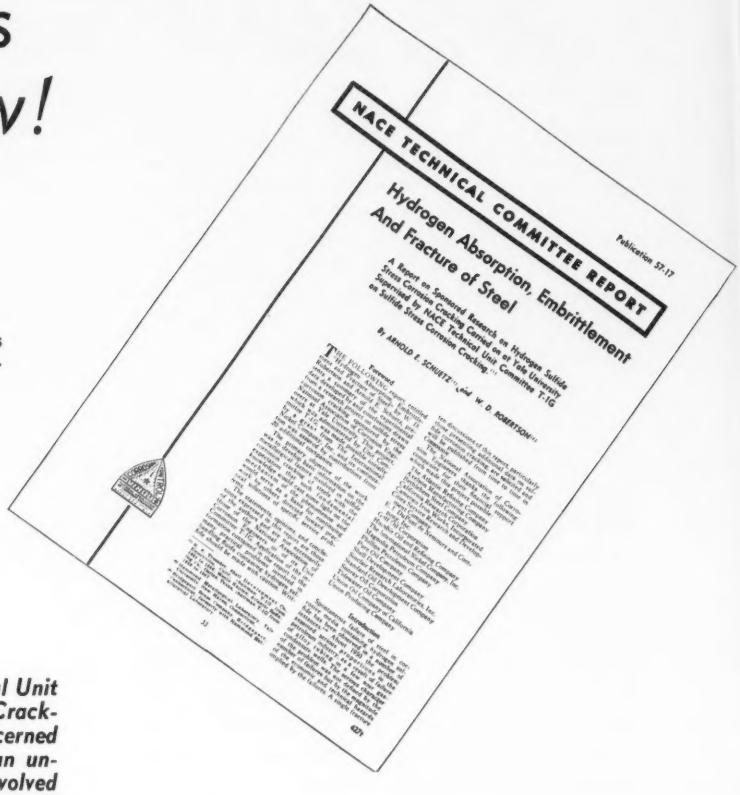
NACE TECHNICAL COMMITTEE REPORT
Publication 57-17
Hydrogen Absorption, Embrittlement And Fracture of Steel
A Report on Sponsored Research on Hydrogen Sulfide Stress Corrosion Cracking Carried on at Yale University Supervised by NACE Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking
by ARNOLD E. SCHUETZ and W. D. ROBERTSON

Basic Facts on Mechanism of Hydrogen Sulfide Stress Corrosion

The findings of a 4-year inquiry into the fundamental reactions involved in the so-called spontaneous fracture of steel as a result of hydrogen absorption in the presence of sulfides.

This project, supervised by NACE's Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking, was financed by industrial firms concerned with this problem in the expectation that an understanding of the fundamental reactions involved would lead to a solution.

Data in this report are expected to be useful not only in immediate steps to reduce the losses due to spontaneous failure but also as a foundation for subsequent inquiries into phenomena associated with hydrogen absorption and embrittlement of steel.

To: A. B. Campbell, Executive Secretary
National Association of Corrosion Engineers
1061 M & M Bldg., Houston 2, Texas

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Publication No. 57-17

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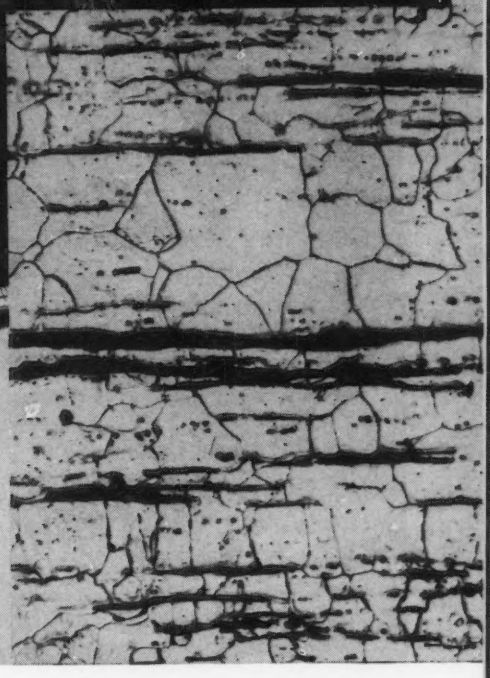

Photomicrograph U-85-3 shows a typical area of the microstructure observed in the subject 5-inch wrought iron pipe.

Metallurgical Report 2187—Underground Oil Line*

This report deals with the metallurgical examination of a length of 5-inch wrought iron pipe, approximately two feet (2′) long, submitted to our laboratory for investigation and comment.

General condition of the as-received sample is shown in large photograph above. Outer surface displayed some few shallow corroded areas but was otherwise in good condition. Inner surface of the pipe sample was also in excellent condition.

ROCKWELL HARDNESS

A cross-sectional ring was prepared for hardness determinations. Hardness values, taken around the cross-sectional ring indicated a B67 hardness typical for wrought iron pipe and in lieu of tensile properties showed that the strength and ductility were likewise normal.

CHEMICAL ANALYSIS

Carbon—.025%; Manganese—.066%; Phosphorus—.230%; Sulphur—.020%; Silicon—.120%; Iron Silicate—2.82%.

SUMMARY

Results of the laboratory examination serve as positive identification of the subject sample as wrought iron. The excellent condition of this sample after more than fifty years of service is noteworthy. This report offers documentary evidence of wrought iron's corrosion control in undergound service. A. M. Byers Company, Clark Building, Pittsburgh 22, Pa.

*Name of actual installation given on request.

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THE NATIONAL ASSOCIATION OF CORROSION ENGINEERS

is a non-profit, scientific and research association of individuals and companies concerned with corrosion or interested in it, whose objects are:

(a) To promote the prevention of corrosion, thereby curtailing economic waste and conserving natural resources.

(b) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

(d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.

(e) To promote standardization of terminology, techniques, equipment and design in corrosion control.

(f) To contribute to industrial and public safety by promoting the prevention of corrosion as a cause of accidents.

(g) To foster cooperation between individual operators of metallic plant and structures in the joint solution of common corrosion problems.

(h) To invite a wide diversity of memberships, thereby insuring reciprocal benefits between industries and governmental groups as well as between individuals and corporations.

It is an incorporated association without capital stock, chartered under the laws of Texas. Its affairs are governed by a Board of Directors, elected by the general membership. Officers and elected directors are nominated by a nominating committee in accordance with the articles of organization. Election is by the membership.

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Corrosion

OFFICIAL MONTHLY PUBLICATION

National Association of Corrosion Engineers

An Association of Individuals and Organizations Concerned with Control of Corrosion

1061 M & M Building Houston 2, Texas

Vol. 14 January, 1958 No. 1

Corrosion Research in Progress

Galvanic Corrosion of Aluminum-Steel And Aluminum-Lead Couples

By M. J. PRYOR*

Introduction

GALVANIC CORROSION, in its various forms, undoubtedly is the major single corrosion problem encountered by the aluminum industry in this decade. It is a matter of concern in marine applications where aluminum superstructures must be joined to steel hulls, in soldered aluminum connections, in stress corrosion problems, in conductor applications, etc. Despite the practical importance of these problems, relatively little basic research into the mechanism of galvanic corrosion of aluminum and its control by means other than by insulation has been carried out.

To remedy this, the author's company some three years ago initiated an extensive investigation of the mechanism of galvanic corrosion of aluminum coupled to a wide variety of dissimilar metals.

It was decided at the outset to determine galvanic current flow between parallel idealized plane electrodes using zero external resistance measuring techniques. It was found to be of advantage to use a glass vacuum-tight cell in which the atmosphere above the solution and solution temperature could be accurately controlled. Current flow measurements of this type could then be correlated with potential, electrode polarization data and weight loss data on the dissimilar electrodes. This enabled the investigator to obtain an overall picture of the reaction mechanism.

*Department of Metallurgical Research, Kaiser Aluminum and Chemical Corp., Spokane, Washington.

Aluminum-Steel Couples

Research thus far has progressed most rapidly in the study of the aluminum-steel and aluminum-lead couples.

The aluminum-steel galvanic couple is widely encountered in practical applications. In chloride containing solutions, aluminum completely protects steel cathodically within the pH range 0-14. The galvanic current and the corrosion rate of the aluminum are at a minimum in the nearly neutral pH range. At these neutral pH values, the rate of galvanic corrosion is controlled by the rate of diffusion of oxygen to the steel cathode. Consequently, the galvanic current and the corrosion rate of the aluminum are proportional to the area of the steel cathode and are independent of the area of the aluminum anode. Factors increasing the access of oxygen to the cathode surface, such as stirring and aeration, increase the galvanic corrosion rate proportionately.

Aluminum-Lead Couples

Investigation of the aluminum-lead couple is of interest in connection with the performance of aluminum painted with lead-based paints. Although painting of aluminum with lead-based paints would not be recommended, it nonetheless occurs frequently in the field. For reasons not previously apparent, the performance of aluminum coated in this manner has been extremely variable. The rate of corrosion of aluminum and the galvanic corrosion characteristics of the aluminum-lead couple have been determined in aqueous extracts from metallic lead, litharge, red lead and basic lead carbonate. The last three compounds are commonly used pigments in lead-based paints. Only in metallic lead and litharge extracts were the corrosion and galvanic corrosion of aluminum more severe than that in distilled water and only there was metallic lead deposited from solution onto the aluminum. Both red lead and basic lead carbonate extracts were relatively harmless.

In a lead-based paint, lead pigments reacted with the vehicle to form lead soaps. Lead linoleate, ricinoleate and laurate would be typical of the soaps formed in lead-based alkyd paints. Aqueous extracts from lead soaps effectively inhibit the corrosion and galvanic corrosion of aluminum, an effect which also has been observed on steel. However, inhibition occurs only in the absence of chloride or sulfate ions and the presence of even 5 ppm chloride ions causes severe corrosion. These observations indicate that if chloride or sulfate ions are kept from the paint-metal interface, paints pigmented with red lead or basic lead carbonate will be compatible with aluminum. When small amounts of chloride permeate the paint coating, the passivity of the aluminum will be broken down and severe corrosion, and also galvanic corrosion, of the aluminum will occur.

The two phases of the work on galvanic corrosion outlined above constitute only a portion of the overall planned investigation. However, by building up a store of basic information of this type, the problem in its various facets will become more subject to effective control in the field.

TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

The Oxidation of Molybdenum*

By E. S. JONES, CAPT. J. F. MOSHER, RUDOLPH SPEISER and J. W. SPRETNAK

Abstract

Scale formed upon molybdenum consists of either two or three oxide layers., depending upon temperature and time. The outermost layer is the volatile MoO_3. The inner layers of oxide have a composition very close to MoO_2 but do not exhibit an X-ray pattern corresponding to MoO_2.

The rate of evaporation of MoO_3 and the mechanism of molybdenum oxidation were determined. It was found that after an initial parabolic dependence of weight change upon time the rate of oxidation became linear. This is due to the fact that the outer MoO_3 layer is non-protective so that the rate of oxidation of the suboxide to the trioxide at the suboxide-trioxide interface is equal to the rate of diffusion of oxygen through the suboxide film to the metal-suboxide interface. It was also found that the presence of liquid MoO_3 increased the rate of oxidation of molybdenum.

6.3.9

Introduction

A STUDY of the oxidation of molybdenum is complicated by the fact that many oxides of molybdenum have been shown reliably to exist. The compounds of molybdenum and oxygen reported to date[1-8] are Mo_2O, MoO_2, Mo_4O_{11}, Mo_8O_{23}, Mo_9O_{26}, $Mo_{10}O_{29}$, $Mo_{11}O_{32}$, and oxides of molybdenum, MoO_3, and Mo_xO_{2x}. Many of these oxides are part of a homologous series[9, (1)] with the general formula Mo_xO_{3x-1}. However, research workers[10, 11] investigating the nature and composition of the oxide scales and films forming on molybdenum at moderate and elevated temperatures have reported only MoO_2, MoO_3, and several oxides[12] whose compositions lie between MoO_2 and MoO_3 and which may well be part of the series Mo_xO_{3x-1}.

Molybdenum dioxide[13,14] (MoO_2) has been described as a brownish-black powder, as a violet crystalline powder, and as a substance forming dark bluish-violet prisms which appear pale violet-red in transmitted light. The density of MoO_2 lies between the values 6.34 and 6.47. The crystal lattice is of the rutile type with two molecules in the unit cell.[15] It is reduced by hydrogen to the metal without the formation of intermediate oxides. It is insoluble in cold or hot water and slightly soluble in hot concentrated sulfuric acid.

Molybdenum trioxide[14] (MoO_3), the end product of the complete oxidation of molybdenum is a stable white powder which often has a tinge of blue or green due to the presence of minute amounts of metal ions in lower valence states.

At red heat, MoO_3 turns yellow, but upon cooling its original color is restored. MoO_3 is decidedly volatile, fuming visibly at 1292 F (700 C).

The vapor pressure of MoO_3 as given by Kelly[14a] is for the reaction:

$$MoO_3\,(s) = MoO_3\,(g)$$

$$\log\ P(\text{atm}) = \frac{-15{,}110}{T} + 1.46\ \log\ T - 1.32 \times 10^{-3}\,T + 9.071$$

and for the reaction:

$$MoO_3(1) = MoO_3(g)$$

$$\log P(\text{atm}) = \frac{-11{,}280}{T} - 7.04 \log T + 30.494$$

The melting point of MoO_3 is 1462 F (795 C);[16] its density at room temperature is 4.692. Sublimed MoO_3 forms rhombic tablets and needles and X-ray studies[17,18] indicate that the unit cell contains four molecules of MoO_3 The stoichiometric formula for MoO_3 in the vapor phase[5] is Mo_xO_{3x} where x can have the value 3 and 5.

Quantitative study of the oxidation of molybdenum has been pursued by a number of investigators.[10, 11, 12, 19, 20, 21, 22] Gulbransen and his co-workers[10, 11] measured the rate of oxidation in the pressure range 10^{-6} to 76 mm Hg of oxygen. A substantial increase in oxidation rate was observed with an increase of either temperature or pressure. At low pressures and times less than an hour, molybdenum oxidizes according to a parabolic rate law. Below 842 F (450 C), the oxide formed as a thin surface film is MoO_3; above 842 F (450 C), MoO_2 forms with an associated increase in oxidation rate; with extended test times the oxidation curve deviates from the parabolic, although the coatings remain partially protective. The rate constants[11] for the parabolic oxidation of molybdenum, tungsten and iron at 7.6 cm of oxygen pressure and 450 C are 3.33×10^{-13}, 1.67×10^{-13} and 0.334×10^{-13} (g per sq cm) per sec, respectively.

Peterson and Fassell[21] studied the oxidation of molybdenum at high pressures (1 to 47.6 atm oxygen) in the temperature range 932 F (500 C) to 1292 F (700 C). The only oxidation product observed under these conditions is MoO_3. Above 976 F (525 C) the oxidation rate becomes sensitive to pressure, increasing with an increase in pressure; at nearly all temperatures and pressures the oxidation is linear, indicating a non-protective coating. However, at temperatures between 1202 F (650 C) and 1292 F (700 C) a parabolic oxidation rate was observed by these investigators at low pressures, changing gradually to a linear rate at higher pressures.

Lustman[19] reports that data prior to 1950 on the oxidation of molybdenum in air as a function of temperature are scanty. He noted that the oxidation rate not only increases with temperature but that during the course of a test at a given temperature, it also may increase with time. This phenomenon may correspond to the "self-heating" due to the exothermic heat of reaction as noted by Peterson and Fassell[21] at 1292 F (700 C). Lustman also reported that above the melting point of molybdenum trioxide, 1462 F (795 C), an essentially constant rate of oxidation is observed.

Simnad and Spilners[22] have studied the mechanism of the oxidation of molybdenum in pure oxygen at one atmosphere in the temperature range 932 F (500 C) to 1292 F (700 C). They found that the vaporization of MoO_3 from the surface of an oxidizing specimen into pure oxygen at 1 atmosphere is linear with time, with an activation energy of 53,000 cal per mol below 1202 F (650 C) and 89,600 cal per mol at temperatures above 1202 F (650 C). Simnad and Spilners found that at low temperatures the formation of MoO_3 is parabolic and at higher temperatures is linear. The suboxide MoO_2 thickness does not increase beyond a very small critical thickness. At temperatures above 1327 F

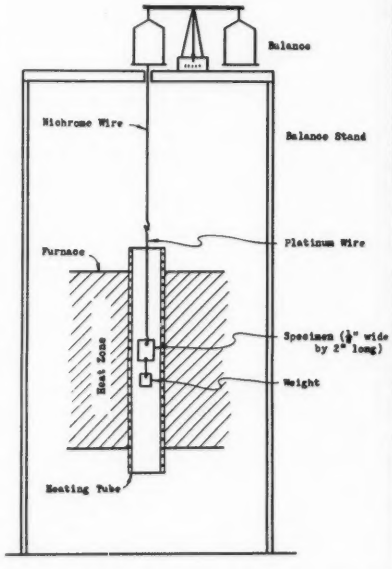

Figure 1—Schematic diagram of apparatus used for measuring oxidation rates.

★ Submitted for publication April 26, 1957. A paper presented at the Thirteenth Annual Conference, National Association of Corrosion Engineers, St. Louis, Missouri, March 11-15, 1957.

(1) Magneli and others at the University of Upsala, Sweden have demonstrated the existence of a number of oxides with the stoichiometric formula Mo_xO_{3x-1} between the oxides MoO_2 and MoO_3. The Mo_xO_{3x-1} compounds are composed of tetrahedra and octahedra of oxygen atoms with a Mo atom in the center (valence 4 or 6) with these structural units being combined in varying proportions with varying numbers of "edge" or "vertex" joining (i.e., greater or lesser sharing of oxygen atoms between the tetrahedra and octahedra). Generally the unit cells are large and have low symmetry.

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Speiser

Mosher

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Spretnak

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(725 C) catastrophic oxidation of an autocatalytic nature was encountered by them.

This research reports the results of extensive studies made of the oxidation of molybdenum in air.

Apparatus and Experimental Procedure

Molybdenum samples in the form of sheet and wire were heated in still air and in metered moving air. The specimens suspended by a platinum wire from one pan of a chainomatic balance (with a sensitivity of 0.05 mg) were weighed at intervals during a run (see Figure 1). The air passed over the sample was first dried by passing through drying towers containing anhydrous calcium sulfate and phosphorous pentoxide and then preheated in a lower part of the furnace to the temperature of the sample before being passed over the sample. The temperature of the sample was measured with a platinum-platinum, 10 percent rhodium thermocouple and the temperature of the sample was maintained constant to ± 3.6 F (± 2 C).

Commercially pure wrought and annealed molybdenum sheet, 2 inch x ½ inch x 0.010 inch, and wire specimens, 30 and 50 mils in diameter and 1¾ inches long were used for the oxidation experiments. The chemical composition of the sheet and wire was molybdenum, 99.9 percent; iron 0.005 percent; carbon 0.003 percent. These samples were prepared for oxidation measurements by the following procedure:

1. Pickle in nitric acid (30 percent by weight HNO_3).
2. Wash in a stream of distilled water.
3. Wash in boiling 1 normal NH_4OH.
4. Wash in distilled water.
5. Store under acetone until ready for testing.

At all times the specimens were handled with tweezers to prevent surface contamination. Treatment of the samples by the above procedure yielded samples which gave reproducible rates of oxidation.

Cast cylinders of pure solid MoO_3 (analytical grade, anhydrous MoO_3) 1 inch diameter x 1½ inches long were used to determine the rate of sublimation of MoO_3. The samples were suspended with a platinum wire tied around the sample. The MoO_3 sublimation rates were measured with the same equipment used to measure rates of oxidation, the weight loss of the specimen being measured at time intervals at constant temperature.

At those temperatures where the evaporation rate of MoO_3 was appreciable, it was necessary to remove condensed MoO_3 from the suspension wires before weighing. This was accomplished by a small loop fashioned from platinum wire which could be raised and lowered from the top of the reaction tube to the sample. The loop encircled the suspension wire and satisfactorily removed any MoO_3 crystals that were clinging to the suspension wires. On long time tests, eight or more hours, the sample and wire could be removed and cleaned and replaced in the reaction tube in less than 10 seconds, which was a small period of time compared to the duration of an oxidation test.

A technique had to be developed for the preparation of the molybdenum oxide scale for metallographic examination. The commercial resin "Vibrin 108" which in the uncured form is liquid and is hardened through the addition of an accelerator and a catalyst, without requiring the use of heat or pressure, was used to mount the specimens. In this way the brittle and fragile oxides at the surface of the sample were not damaged during the mounting and polishing operation.

Considerable polishing experimentation was required to evolve a successful technique to prepare specimens for satisfactory metallographic examination. The polishing procedure finally adopted was as follows:

1. After being mounted, specimens were lightly rough polished on a No. 120 grit abrasive belt (dry).
2. Specimens were hand polished on emery papers, 1/0 through 3/0. Only a few strokes were necessary on each paper to change the direction of the scratches resulting from the previous paper.
3. The specimens were final polished on a high speed wheel covered with "Miracloth." Rouge was used as the polishing agent. A few drops of an

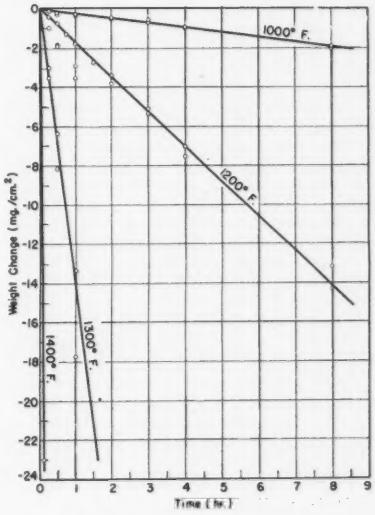

Figure 2—Rate of sublimation of MoO_3 at several temperatures.

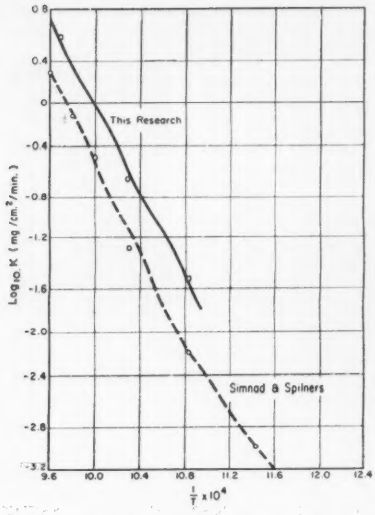

Figure 3—Plot of logarithm of the rate of sublimation of MoO_3 versus the reciprocal of the absolute temperature.

TABLE 1—Rates of Sublimation of MoO_3

Temperature, Degrees F	Temperature, Degrees C	k_3, mg/cm²/Min.
1000	538	0.00341
1200	649	0.0292
1300	704	0.237
1400	760	3.710

aqueous solution of chromic acid (10 percent) and phosphoric acid (4 percent) were applied intermittently during the operation. This solution acted as a mild etchant on the unoxidized portion of the molybdenum specimen and revealed its structure clearly without noticeable effect on the oxide layers. Water was added sparingly to the wheel, previous experience having shown that water in large quantities tended to loosen and remove oxides from the surface of the specimen.

Results and Discussion

Sublimation of MoO_3. Reliable expressions for the kinetics of oxidation of molybdenum cannot be made without an accurate estimate of the MoO_3 volatilized during an oxidation experiment. Consequently the rate of sublimation of MoO_3 into air at several temperatures was measured on cast specimens of pure MoO_3 by procedures described in a previous section of this paper. The results of these experiments are given in Figure 2 and Table 1. Obviously, the rate of sublimation is constant at all temperatures as is expected.

The logarithm of the rate constant is plotted against the reciprocal of the absolute temperature in Figure 3. The data in this research are compared with the data of Simnad and Spilners.[22] A break in the curves of both researches seems to occur at about 1202 F (650 C). Above 1202 F (650 C) the activation energy of sublimation as measured in this research is 85,800 cal per mol; this is to be compared to 89,600 cal per mol measured by Simnad and Spilners. The agreement is excellent considering the difference in the two methods of determining the rates of sublimation. Simnad and Spilners estimated the rate of vaporization directly from oxidizing molybdenum samples by making a material balance and computing the amount of MoO_3 which must have oxidized away, whereas in this research, the sublimation rate of pure solid MoO_3 was directly measured. Below 1202 F (650 C) the sublimation activation energy is much lower; 53,000 cal per mol as measured by Simnad and Spilners and 50,800 cal per mol, in the range 887 F (475 C) to 976 F (525 C), as reported by Gulbransen. A change in the mechanism of vaporization of MoO_3 must take place at about 1202 F (650 C). This change is possibly due to a crystal structure change that MoO_3 undergoes in the neighborhood of this temperature since it is observed that MoO_3 turns from white to yellow upon heating to red heat and then turns from yellow to white again upon cooling. The explanation offered by Simnad and Spilners that this change in the rate of sublimation of MoO_3 is caused by a change in the oxidation mechanism of sublimation of MoO_3 cannot be correct, since this change is observed with pure MoO_3. The converse is of course possible (i.e., that the change in mechanism of sublimation can lead to a change in mechanism of oxidation of molybdenum.)

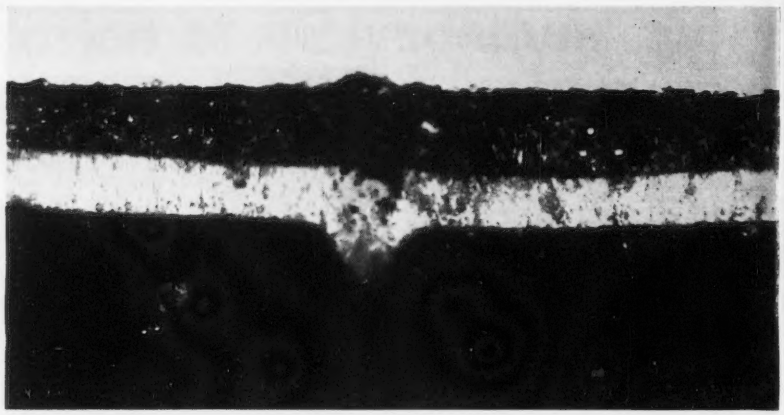

Figure 4—Sheet specimen oxidized 50 minutes at 1060 F (571 C). Section was taken parallel to rolling direction showing layer of MoO_3 and layer of oxide "B" in contact with metal surface. Small rupture in surface of MoO_3 layer is evident. Original magnification, 600X; actual magnification after reduction for engraving purposes, 465X.

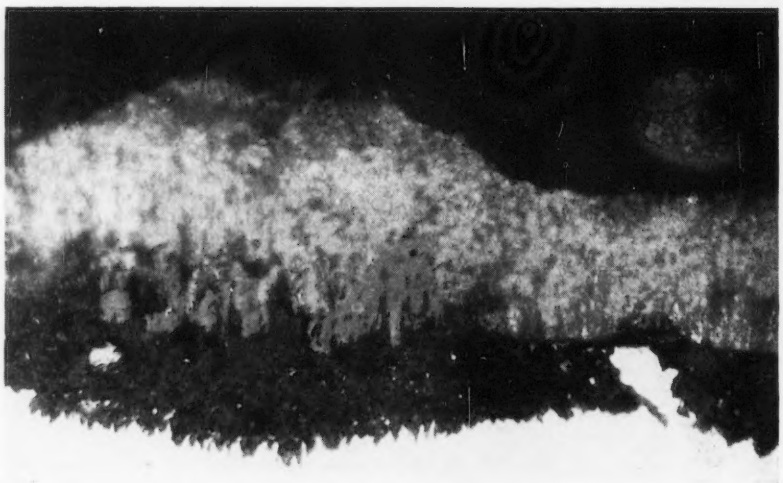

Figure 5—Sheet specimen oxidized four hours at 1060 F (571 C). MoO_3, oxide "A" and oxide "B" apparently coexist at this stage of oxidation. Original magnification, 600X; actual magnification after reduction for engraving purposes, 450X.

Metallographic Observations. The oxidation of molybdenum from the surface inward is a rate process and not an equilibrium process, therefore it would be expected that several layers of oxide would coexist on the molybdenum surface. Surface phases such as:

Molybdenum metal	Molybdenum saturated with oxygen	lower oxide or oxides of molybdenum	MoO_3

can be inferred from the gradient of oxygen activity. A molybdenum sheet sample that has been oxidized 50 minutes at 1060 F (571 C) is shown in Fig-

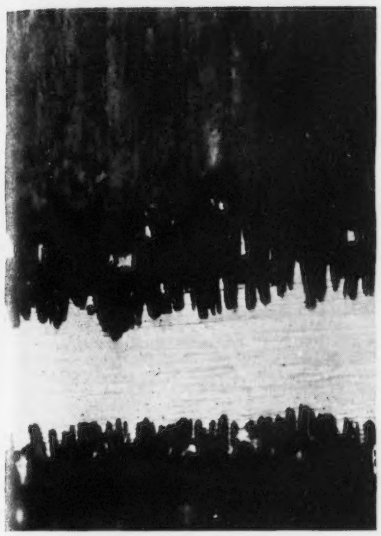

Figure 6—Metal-oxide interface of sheet specimen oxidized for 48 hours at 1060 F (571 C). Original magnification, 500X; actual magnification after reduction for engraving purposes, 280X.

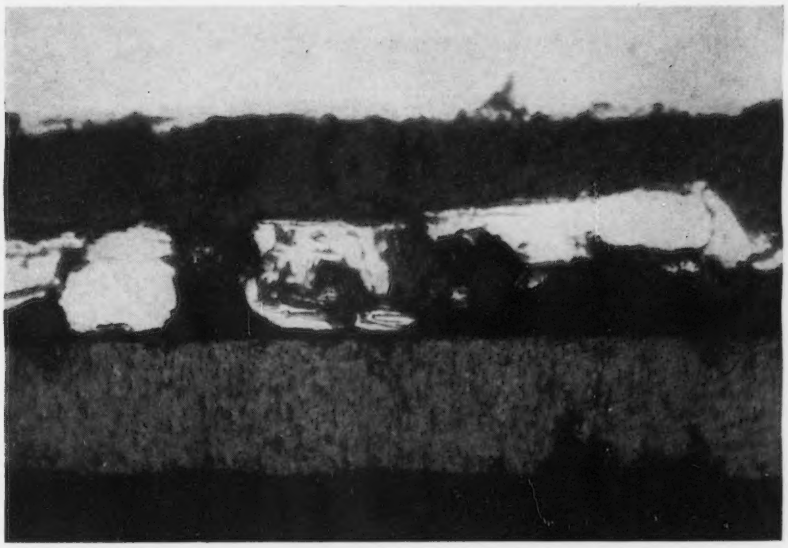

Figure 7—Sheet specimen oxidized two hours at 1060 F (571 C). Section was taken perpendicular to rolling direction showing MoO_3 and layer of oxide "B" surrounding fragments of metal. Original magnification, 600X; actual magnification after reduction for engraving purposes, 560X.

ure 4. The oxide on the surface of the specimen is made up of at least two separate phases. The outer oxide phase (light scale) is MoO_3; oxide "B" (MoO_z where $3 > z > 2$) is adjacent to the unoxidized metal core. A small rupture can be seen in the surface of the MoO_3 scale. Immediately under this break, one can see a small crystal of oxide "A" ($MoO_{z'}$ where $3 > Z' > Z$). Initially when the MoO_3 oxide is thin and coherent so that it is protective and the available oxygen concentration under the MoO_3 is low, oxide "A" is formed. However, when the MoO_3 layer thickens and cracks or if the oxide film is defective due to some other cause such as an eruption in the film as shown in Figure 4, more oxygen is available and oxide "B" is formed. A sheet specimen oxidized for four hours at 1060 F (561 C) is shown in Figure 5. Oxide "A" and oxide "B" and MoO_3 coexist at this stage of the oxidation and it is clear that oxide "B" is in the region of higher oxygen concentration. After 48 hours of oxidation at 1060 F (571 C), oxide "B" is completely consumed and transformed into oxide "A"; this behavior can be deduced from Figure 6.

The operation of rolling molybdenum sheet not only leads to orientation effects, but also leads to some heterogeneity in the oxidation of the sheet. This effect is evident in Figure 7 in which a section perpendicular to the rolling direction shows MoO_3 and a layer of oxide "B" surrounding slivers of unoxidized metal. This is probably due to impurities picked up in the surface of the metal during rolling. Figure 8 is a low magnification micrograph of the surface of the oxide formed after short oxidation times. Small, pyramid-shaped "eruptions" may be seen to occur on the surface of the MoO_3 layer. In addition, a dark streak parallel to the direction of rolling of the sheet is present which is free of MoO_2. These streaks are visually apparent on the surface of specimens oxidized for short periods of time and gradually disappear as the time of exposure to oxidation is increased. (This behavior can be eliminated if the procedure for sample preparation for oxidation rate measurements is followed as described in the previous section.)

A photograph of the general appearance of the sheet specimens before and after oxidation is included in Figure 9. In specimen C, the yellowish outer layer of MoO_3 has been removed by immersion of the specimen in concentrated NH_4OH, revealing the dark inner layer of oxide "A". A photomicrograph of a sheet specimen from which the MoO_3 layer has been stripped is shown in Figure 10. Crystals of adherent, columnar oxide "A" remain on the specimen and the finger-like projection of this oxide into the metal is clearly shown at high magnification in Figure 11.

The structure of the scale on molybdenum sheet oxidized at 1150 F (621 C) for four hours is shown in Figure 12. The pronounced granular appearance of the outer layer of MoO_3 indicates that crystal growth in the MoO_3 phase has occurred at this temperature resulting in a porous MoO_3 oxide in contrast to the dense and more protective MoO_3 formed at lower temperatures.

X-ray Diffraction Studies of the Oxidation Products

The yellowish outer oxide formed on oxidized specimens was identified as being molybdenum trioxide (MoO_3). Positive identification was made by comparison with patterns obtained from chemically pure molybdic anhydride and by comparison with diffraction data given for the oxide by ASTM standard card No. 1-0706. The sublimed yellowish-white crystal condensate deposited upon the furnace walls was also identified as MoO_3. Diffraction patterns obtained from the bluish-black, columnar oxide crystals (oxide "A") have not been identified as yet as to composition. X-ray patterns of this oxide while still adhering to the metallic substrate (after the removal of the MoO_3 layer with concentrated NH_4OH) reveal the presence of weak molybdenum lines which may be due to the presence of unoxidized metal which are entrapped during the growth of the suboxide (see Figure 7). Photograms of this oxide after mechanical removal from the specimen also reveal the presence of weak molybdenum lines, thus eliminating the possibility of these lines being obtained from the substrate.

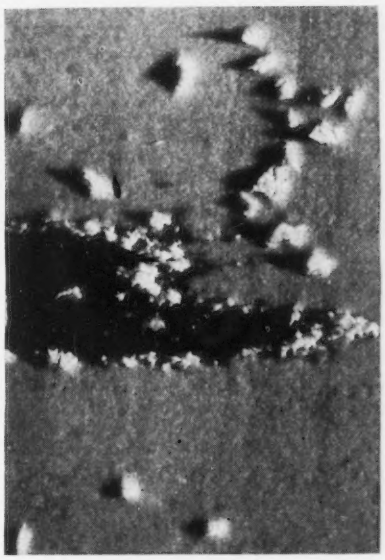

Figure 8—Photomicrograph of the surface of a sheet specimen oxidized 50 minutes at 1060 F (571 C). Small "eruptions" on the surface of MoO_3 are evident as is a dark streak parallel to the rolling direction. Oblique lighting. Original magnification, 100X; actual magnification after reduction for engraving purposes, 50X.

The patterns for oxide "A" do not check with those reported for molybdenum dioxide (MoO_2) (ASTM card No. 2-0422) nor could the data given by Hickman and Gulbransen[10] be correlated with the present results. Hagg and Magneli[2,3,4,5,6] report a number of suboxides of the type Mo_nO_{3n-1}, but unfortunately they do not report the "d" spacings. Thus intercomparisons cannot be made with the suboxides found in this research.

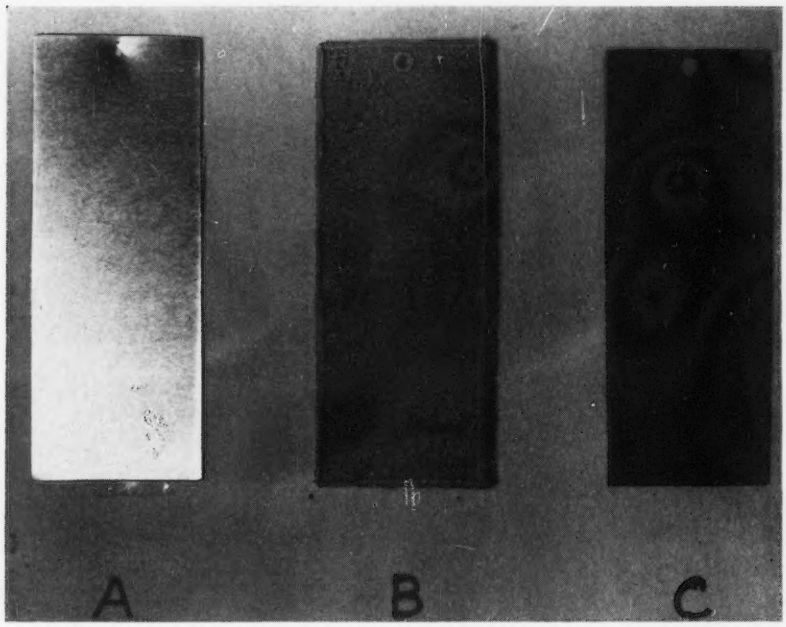

Figure 9—A—sheet specimen before oxidation; B—specimen oxidized 20 hours at 1060 F (571 C); C—specimen oxidized 20 hours at 1060 F (571 C). Outer layer was removed by immersion of specimen in concentrated NH_4OH.

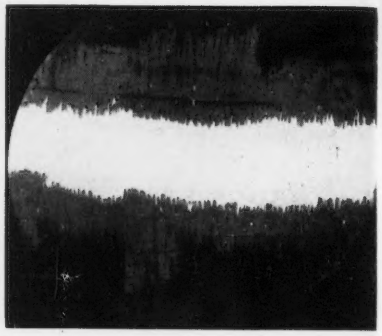

Figure 10—Sheet specimen, oxidized 20 hours at 1060 F (571 C), from which the MoO_3 layer has been removed by immersion of the specimen in NH_4OH. Crystals of oxide "A" remain on specimen. Original magnification, 250X; actual magnification after reduction for engraving purposes, 110X.

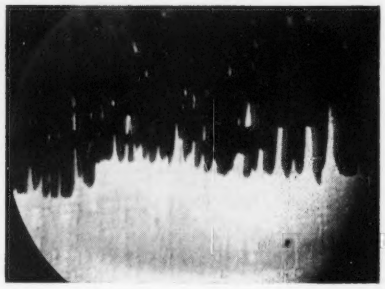

Figure 11—High magnification view of columnar crystals of oxide "A" showing penetration into metal surface. Sheet specimen was oxidized 20 hours at 1060 F (571 C). Original magnification, 1080X; actual magnification after reduction for engraving purposes, 400X.

Figure 12—Oxide formation on a specimen oxidized four hours at 1150 F (621 C). Crystals of MoO_3 on the surface have a markedly granular appearance. Original magnification, 600X; actual magnification after reduction for engraving purposes, 240X.

Diffraction patterns which represent oxide "B" have been obtained. However, attempts to completely separate oxide "B" from oxide "A" have been unsuccessful and efforts to obtain a sharp diffraction pattern from the black, finely granular oxide "B" in contact with the metallic substrate also have been unsuccessful.

A diagram showing the relative line spacings and intensities obtained from various diffraction patterns is shown in Figure 13. It can be seen that neither oxide "A" nor oxide "B" have patterns even remotely resembling MoO_2. In fact, MoO_2 was never observed in any of the many samples of molybdenum that were oxidized in air in this laboratory. MoO_2 (in appreciable quantities) has been found in this laboratory[23] only in ternary systems such as Mo-Ni-O. It should be mentioned that in principle a MoO_2 layer should exist adjacent to the metal, but apparently this layer is too thin to detect.

Examination of back reflection X-ray diffraction patterns obtained from the surface of sheet specimens indicate that a condition of preferred orientation exists, the pole figures in general being in agreement with the results reported by Semschyshen and Timmons.[24] This condition is believed to influence the formation of the dark streaks that are visible on the surface, parallel to the direction of rolling (Figure 8), of specimens oxidized for short periods of time in the temperature range of 975 F (524 C) to 1150 F (621 C). Segregated impurities in molybdenum or dirt on the surface of the rolls which become elongated in the direction of rolling also may contribute to the occurrence of these small areas which seem temporarily to resist oxidation.

Rate Measurements. Oxidation tests on molybdenum sheet were carried out by the methods described in the previous section. A log-log plot of weight gain versus time for the oxidation of molybdenum sheet at 975 F (524 C) is shown in Figure 14. (The log-log plot is used for convenience.) Examination of the rectilinear plot shows that initially the oxidation curve is parabolic followed by an interval where the rate of oxidation increases to a constant rate which then persists to the end of the test. This behavior can be interpreted if it is assumed that initially the suboxide "B" and the outermost oxide scale MoO_3 are protective so that the observed weight gain is parabolic; then as the oxidation proceeds, oxide "B" is converted into oxide "A" which is less protective (i.e., oxygen ions diffuse more rapidly through oxide "A" than through oxide "B"). MoO_3 ceases to be protective because it has thickened to a point where it is cracked and fissured. Consequently, the rate of oxidation is constant after 20 hours. Oxide "A" is converted into non-protective MoO_3 at a constant rate because the partial pressure of oxygen is constant. MoO_3 evaporates at a constant rate. Futhermore, since oxide "A" can form only at concentrations of oxygen above a critical concentration, the path length for diffusion of oxygen is constant, hence the rate of formation of oxide "A" is constant.

The rate of oxidation of molybdenum sheet, corrected for the MoO_3 volatilized away during oxidation tests at various temperatures, is shown in Figures 15 and 16. In all cases it is seen that the latter part of the oxidation curve is linear.

Molybdenum trioxide melts at 1463 F (795 C) and the eutectic[25] MoO_2-MoO_3 melts at 1432 F (778 C); therefore, at 1500 F (816 C) the oxidation of molybdenum occurs in the presence of liquid MoO_3 and the liquid eutectic mixture. It may be observed in Figure 17 that only 0.5 percent of the total oxide formed (at 1500 F) (816 C) remains on the surface of the specimen at the conclusion of the test, the remainder having volatilized away. Compare this figure with results for 1300 F (704 C), 22 percent. Upon withdrawing the speci-

TABLE 2—Oxidation of 0.010 Molybdenum Sheet in Still Air

Temp., Degrees F	Temperature, Degrees C	Relative Rates of Oxidation
1300	701	1
1500	816	42.5
1800	983	39.5

men from the hot zone, the oxide on the surface freezes instantly into a "frost-like" pattern of steel-gray crystals. Oxidation of the specimen at the lower edges where the molten oxide tends to gather is greatly accelerated; thus it seems that the presence of liquid molybdenum oxides greatly accelerate the oxidation as reported by Rathenau and Meijering.[25]

At higher temperatures, i.e. 1800 F (938 C), MoO_3 volatilizes away as rapidly as it is formed; consequently there are no liquid oxides present on the surface and the rate of oxidation of molybdenum is actually slightly lower at this temperature than at 1500 F (816 C). This can be seen clearly in Table 2, where the rates of oxidation relative to the rate at 1300 F (704 C) are listed. It also is evident from these results that the influence of liquid oxides on the oxidation rate of molybdenum is of considerably greater influence than the effects due to the dissociation of MoO_3—since the dissociation of MoO_3 should be greatly increased with an increase in temperature.[26]

Mechanism of Oxidation. To ascertain whether the oxidation of molybdenum proceeded by the migration of molybdenum ions through the oxide to the oxide-air interface or by the diffusion of oxygen ions through the oxide to the metal-metal oxide interface, marker[27,28] experiments with inert chromium oxide (Cr_2O_3) were carried out. A thin coating of Cr_2O_3 was applied in a form of a slurry to molybdenum sheet and wire specimens and allowed to dry before oxidation testing. In all cases it was found that the deep green Cr_2O_3 remained on the outer oxide surface; in no instance was the Cr_2O_3 coating enveloped by the oxide formed as oxidation proceeded. Simnad and Spilners[22] obtained similar results using radioactive silver as a marker. These experiments indicate that the oxidation is initiated and then proceeds at the metal-oxide interface through the diffusion of oxygen ions ($O^=$) to the metal-oxide interface.

Further evidence in support of this assumption can be obtained by comparing the oxidation of iron wire to the oxidation of molybdenum wire. In the case of iron, it is well established that iron ion diffusion through FeO to the FeO/Fe_3O_4 interface[2] controls the oxidation of iron.[29] A dramatic illustration of this difference in mechanism is illustrated in Figures 18 and 19 which compare a partially oxidized molybdenum and iron wire. It can be seen that the outer MoO_3 oxide is split and fissured and is no longer protective. A coherent suboxide layer surrounds the sound molybdenum metal core. In contrast, it is evident that the metal in the iron core has been depleted, the migration of the iron ions through the oxide leading to a void in the iron core through the condensation of vacancies.

Quantitatively the oxidation of molybdenum can be described as follows,

[2] In the Fe_3O_4 both iron ions and oxygen ions are diffusing, whereas in the outermost Fe_2O_3 scale, oxygen is rapidly diffusing to the Fe_3O_4/Fe_2O_3 interface.

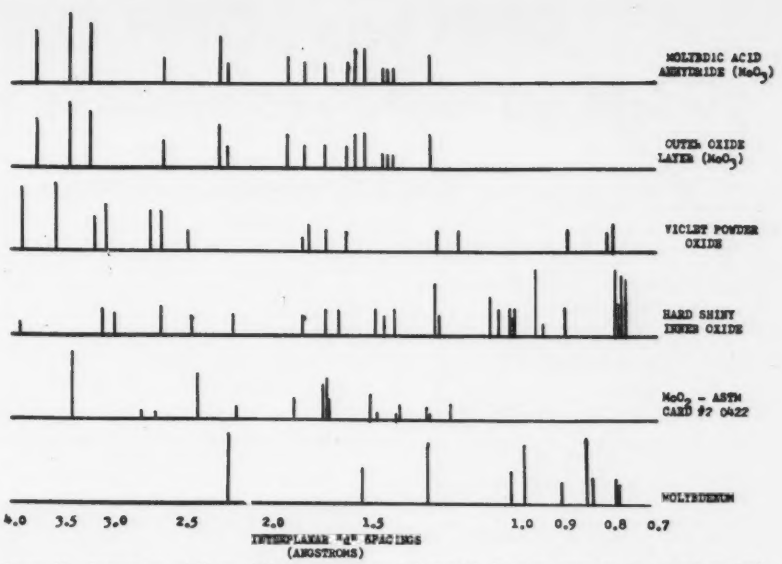
MOLYBDIC ACID ANHYDRIDE (MoO_3)
OUTER OXIDE LAYER (MoO_3)
VIOLET POWDER OXIDE
HARD SHINY INNER OXIDE
MoO_2 - ASTM CARD #7 0422
MOLYBDENUM
4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.9 0.8 0.7
INTERPLANAR "d" SPACINGS (ANGSTROMS)

Figure 13—Comparison of X-ray diffraction patterns (schematic). Relative intensity is plotted vertically.

Rate of increase of sample weight = (Rate of formation of suboxide, MoO_z)f_1 + (Rate of formation of MoO_3 from MoO_z)f_2 — (Rate of sublimation[3] of MoO_3)f_3.

Symbolically, this relation can be written as

$$\frac{dW}{dt} = \frac{dW_{(MoO_z)}}{dt} f_1 + \frac{dW_{(MoO_3)}}{dt} f_2 - \frac{dW_{(MoO_{3\,(g)})}}{dt} f_3 \qquad (1)$$

where $f_1 = \frac{z \times 16}{96 \times 1 + z \times 16}$, $f_2 = \frac{3 \times 16}{96 + 3 \times 16} = \frac{1}{3}$ and $f_3 = 1$.

At constant temperature, the rate of sublimation of MoO_3 is constant; therefore, $dW_{(MoO_{3\,(g)})}/dt = k_3$, where k_3 is a constant. In the region where the oxidation curve is linear, the outer MoO_3 oxide layer is non-protective. Therefore the partial pressure of oxygen is constant at the MoO_z/MoO_3 interface, and since the MoO_z concentration also does not change at this interface, $d(MoO_3)/dt = k_2$, k_2 being a constant.

The partial pressure of oxygen must fall from its value at the MoO_z/MoO_3 interface to that of the dissociation pressure of MoO_z at the Mo/MoO_z interface. The thickness of the MoO_z layer depends upon the rate of diffusion of the oxygen ion through the suboxide and if the assumption is made that the rate of oxidation of molybdenum to the suboxide is fast compared to the rate of diffusion of the oxygen ion, the thickness of the suboxide will remain constant during an oxidation test at a fixed temperature. This means that the path length for diffusion of the oxide ion will be constant. This behavior is observed experimentally—consequently when the oxidation curve becomes linear it means that a steady state with respect to the MoO_z concentration must be established and therefore $dW_{(MoO_z)}/dt = 0$.

[3] Although a MoO_2 oxide layer has not been observed in metallographic examination, a layer of MoO_2, too thin to be observed, must be present in the scale. This is the case even after very long periods of oxidation, consequently it would cause little error to omit MoO_2 from consideration in equation 1.

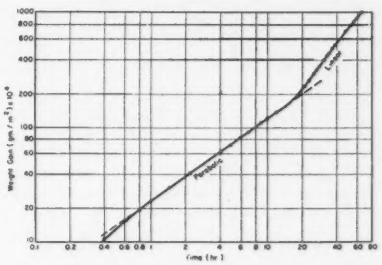

Figure 14—Log-log plot of the oxidation of 10-inch fansteel molybdenum sheet in still air at 975 F (524 C).

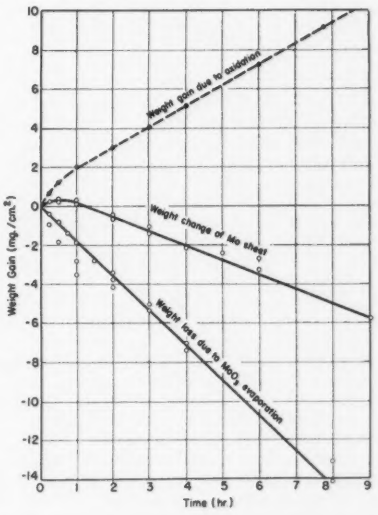

Figure 15—Rate of formation and evaporation of MoO_3 at 1200 F (649 C).

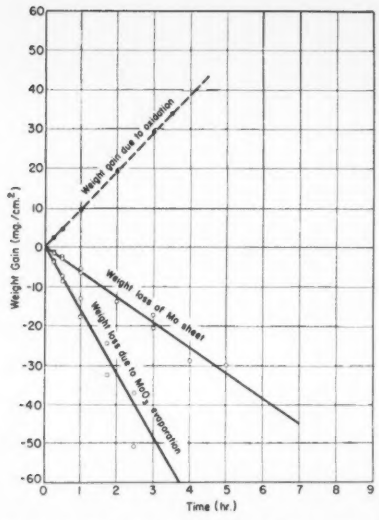

Figure 16—Rate of oxidation of molybdenum sheet at 1300 F (704 C).

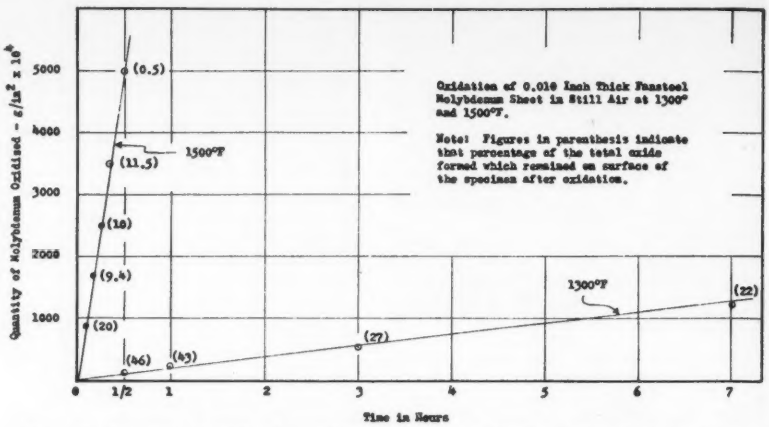

Figure 17—Percentage of total oxide remaining on sample as a function of time and temperature.

As a result of these considerations, equation (1) can now be written as

$$\frac{dw}{dt} = f_2k_2 - f_3k_3 \qquad (2)$$

which is the equation of a straight line and conforms to the experimental behavior exemplified in Figures 15 and 16.

The first part of the oxidation curve has a form that indicates that the initial rate of oxidation of molybdenum is a function of the thickness of the suboxide and MoO_3 scales. The initial form of the curve in Figure 14 can be taken into account in equation (5) if a term involving the conversion of MoO_x into MoO_x' (conversion of oxide "B" into oxide "A") is included where the rate constant k_2' is replaced by a rate constant k_2, such that $k_2 > k_2'$.

Summary

1. The oxidation of molybdenum in the temperature range 900 F (482 C) to 1800 F (983 C) has been studied.
2. Three separate oxide phases were observed to exist on the surface of specimens oxidized under certain conditions of time and temperature. The outer oxide is MoO_3; the inner suboxides yield X-ray patterns differing from that for MoO_2.
3. The rate of sublimation of MoO_3 has been determined at several temperatures.
4. Presence of liquid MoO_3 and/or liquid eutectic suboxide-MoO_3 on the surface of an oxidizing specimen greatly increases the rate of oxidation of molybdenum.
5. Initially the oxidation of molybdenum is parabolic changing ultimately to a constant rate. The detailed character of the oxidation curve depends upon time, temperature and sample history.
6. A mechanism for the oxidation of molybdenum is given.

Acknowledgment

The work reported here was carried out under the terms of a contract between The Ohio State University Research Foundation and the Office of Naval Research, Department of the Navy. The authors also wish to acknowledge the invaluable assistance of Dr. William L. Larsen in the success of this research.

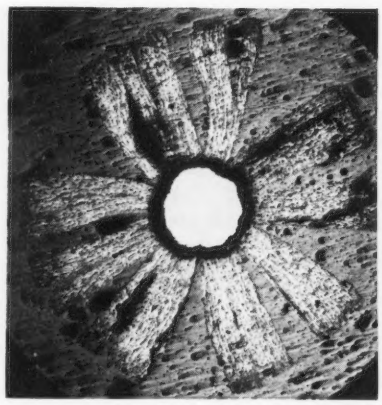

Figure 18—Molybdenum wire (0.050 inch diameter) oxidized 66 hours at 1060 F (571 C). Extreme condition of splitting of MoO_3 layer is shown.

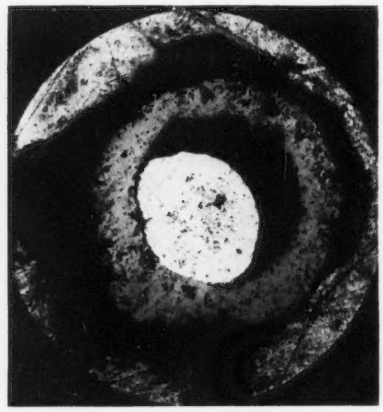

Figure 19—Oxide formed by heating iron wire in dry oxygen for 10 hours at 1500 F (816 C).[29]

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DISCUSSION

Question by Thomas L. Hoffman, Phillips Petroleum Company, Atomic Energy Division, Idaho Falls, Idaho: Can you report the solubility of MoO_3 in chemicals other than NH_4OH solutions?

Reply by Capt. J. F. Mosher:

The solubility of MoO_3 in other solvents was not determined.

The Effect of NO, HNO_2, and HNO_3 On Corrosion of Stainless Steel by H_2SO_4 ★

By W. P. McKINNELL, JR.,[1] L. F. LOCKWOOD,[2] R. SPEISER,[3] F. H. BECK[4] and M. G. FONTANA[5]

Abstract

Earlier work by other investigators has shown that nitrous acid is involved in the passivation of iron in concentrated nitric acid solutions. This paper describes the effect of nitric oxide, HNO_2 and HNO_3 on the corrosion of active Type 302 stainless steel by 10 percent H_2SO_4.

Specimens of Type 302 stainless steel, activated by abrading on 240 grit emery cloth and exposed to 10 percent H_2SO_4 solutions, were passivated by bubbling nitric oxide through the solution. Bubbling oxygen through the solution did not passivate these abraded specimens. Abraded specimens exposed to an atmosphere of nitric oxide before immersion in nitric oxide-free H_2SO_4 remained active. Spot tests of 10 percent H_2SO_4 treated with nitric oxide revealed the presence of nitrite and nitrate ions in the solution. The addition of one part of 10 percent HNO_3 to 60 parts of 10 percent H_2SO_4 was found to produce passivity of abraded stainless steel specimens exposed to the solution. 4.3.2

Introduction

THE ROLE of nitrous acid in the corrosion of iron in nitric acid has been the subject of a number of papers. Bonhoeffer[1] brought out that HNO_2 is involved in the passivation of iron in concentrated HNO_3. The nitrous acid acts as a cathodic depolarizer, and while it may lower the redox potential of the nitric acid slightly, the net effect is to increase the anodic current density to the level required for passivation.

Uhlig and O'Connor[2] also have conducted tests showing that nitrous acid is required in the reactions leading to the formation of the passivating film on iron in nitric acid solutions. They showed that the addition of urea or hydrogen peroxide to nitric acid will prevent the passivation of iron. Urea reduces HNO_2, and H_2O_2 prevents the reduction of HNO_3 to HNO_2.

Stainless steels are passive to solutions of nitric acid over the whole range of concentrations from zero to the fuming grades. However, 18-8 stainless steels may be passive or active in a 10 percent solution of sulfuric acid, depending on the history of the surface of the steel. Pickled surfaces subsequently exposed to air are passive while abraded surfaces are active. This paper describes the effect of nitric oxide, HNO_2, and HNO_3 on the corrosion of Type 302 stainless steel[6] by 10 percent H_2SO_4.

Apparatus and Procedure

The experiments performed in this investigation were of two types: (1) corrosion tests including time-potential measurements, and (2) sorption tests.

The material used in these tests was fully annealed Type 302 strip 0.005 inch thick by one inch wide.[6] Its composition was as follows:

Chromium	18.74 percent
Nickel	8.88 percent
Carbon	0.10 percent
Manganese	1.46 percent
Silicon	0.49 percent
Sulfur	0.018 percent
Phosphorus	0.028 percent

Solution potentials of the stainless steel specimens were measured against a saturated calomel half cell, using the circuit shown in Figure 1. The Leeds and Northrup student type potentiometer, Epply standard cell, Rubicon galvanometer, and calomel half cell were arranged outside an atmosphere box in which the sulfuric acid solution and test specimen were located. The abraded test specimens were finished on 240 grit emery cloth under absolute alcohol, and then stored in alcohol until exposed to the proper atmosphere in the atmosphere box. Pickled specimens were cleaned in absolute alcohol, pickled five minutes in boiling 10 percent H_2SO_4, rinsed in water to remove corrosion products, and then immediately immersed in absolute alcohol for storage. Exposure of both abraded and pickled specimens to air was minimized.

Specimens prepared in this manner were transferred in stoppered tubes of alcohol to the atmosphere box which was then closed and purged of air with the gas to be investigated (O_2 or nitric oxide). Ten percent sulfuric acid solution was saturated with the same gas using a fritted glass bubbler. Several

★ Submitted for publication January 14, 1957. A paper presented at the Thirteenth Annual Conference, National Association of Corrosion Engineers, St. Louis, Missouri, March 11-15, 1957.

(1) Research Engineer, Denver Research Center, The Ohio Oil Company, Littleton, Colorado; formerly DuPont Fellow in Metallurgical Engineering, The Ohio State University.
(2) Dow Chemical Company, Midland, Michigan; formerly Research Fellow, The Ohio State University.
(3) Associate Professor, Department of Metallurgical Engineering, The Ohio State University.
(4) Associate Professor, Corrosion Research Laboratory, Engineering Experiment Station, The Ohio State University.
(5) Professor and Chairman, Department of Metallurgical Engineering, The Ohio State University.

(6) This material was furnished as Type 304 stainless steel but was found to contain 0.10 percent carbon.

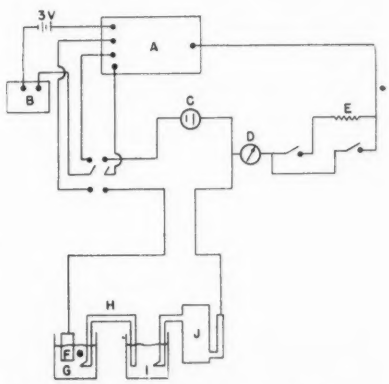

Figure 1—Solution potential measuring equipment. Code to letters is as follows: A—L and N student potentiometer Model No. 7651; B—Variable resistance box; C—Epply standard cell; D—Rubicon light galvanometer catalogue No. 34014; E—10,000 ohm resistor; F—stainless steel specimen; G—Ten percent sulfuric acid solution; H—KCl salt bridge; I—saturated KCl solution; J—saturated calomel half cell.

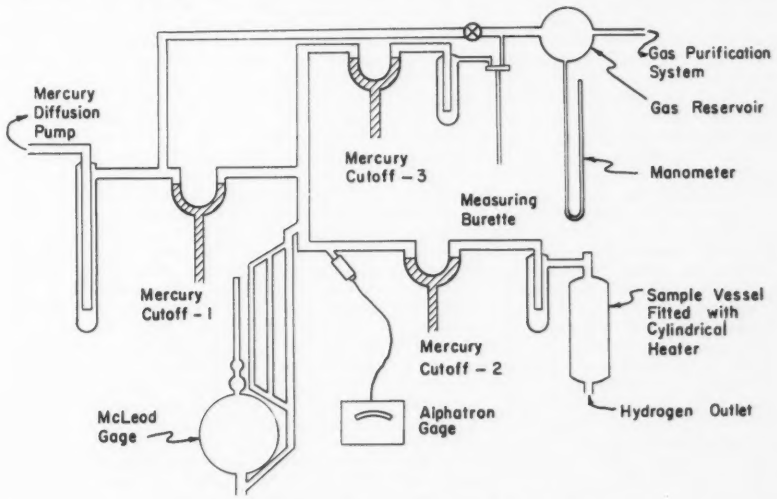

Figure 2—Schematic diagram of sorption apparatus.

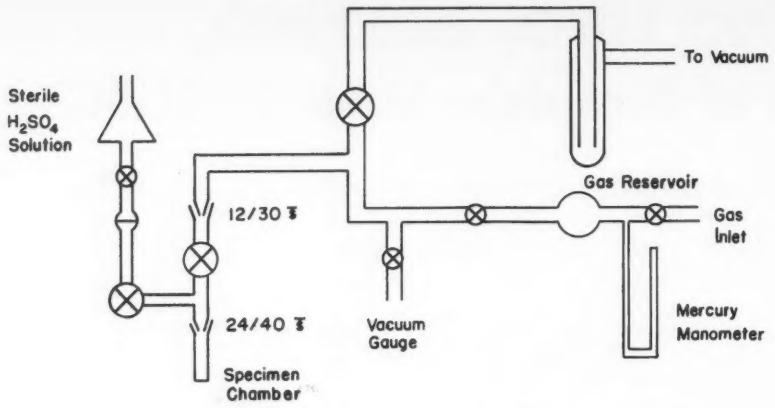

Figure 3—Schematic diagram of corrosion testing apparatus.

TABLE 1—Summary of Corrosion Test Results

Test No.	Solution	Remarks
1	300 ml 10% H_2SO_4	Corroded rapidly
2	300 ml 10% H_2SO_4 + 5 ml 10% HNO_3	Passive, specimen tinted
3	300 ml 10% H_2SO_4 + 10 ml 10% HNO_3	Passive, slight tint
4	300 ml 10% H_2SO_4 + 15 ml 10% HNO_3	Passive, no tint
5	300 ml 10% H_2SO_4 + 50 ml 10% HNO_3	Passive, no tint
6	300 ml 10% H_2SO_4 + 45 ml 10% HNO_3 + 10 grams urea	Corroded
7	300 ml 10% H_2SO_4, N_2 sat'd, NO exposed	Passive
8	300 ml 10% H_2SO_4, N_2 sat'd, NO exposed + 10 grams urea	Corroded

Specimens were of Type 302 stainless steel cut from one-inch strip five mils thick and activated by abrading on No. 120 belt sander. All tests were made in open beakers.

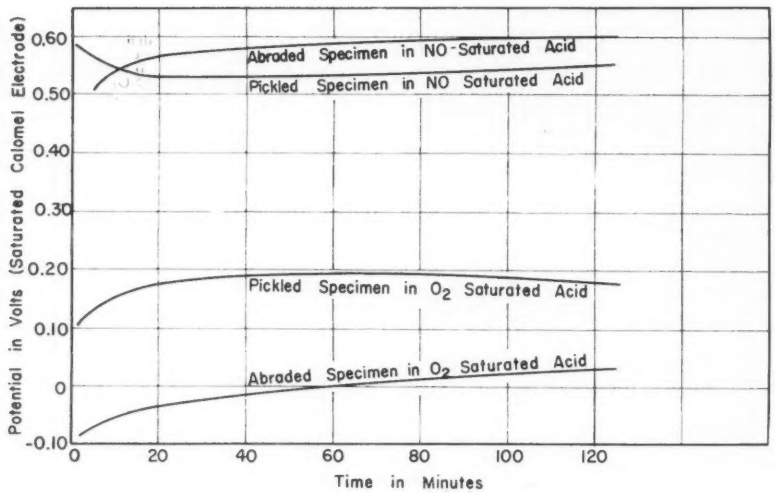

Figure 4—Time-potential curves of Type 302 stainless steel in gas-treated 10 percent H_2SO_4 at 30 C.

hours were allowed for purging the box and saturating the solution.

Oxygen atmospheres were analyzed for nitrogen and nitric oxide atmospheres were analyzed for oxygen to ensure a minimum of contamination. The specimens were removed from their tubes and immersed in 10 percent sulfuric acid when gas analyses showed the box to be satisfactorily purged. Tank oxygen was used after purification by a train consisting of concentrated H_2SO_4, platinized asbestos at 600 C, and two towers of magnesium perchlorate. The nitric oxide was used as it came from the tank, without further purification.

To test the effect of nitric oxide on distilled water and on sulfuric acid solutions, the following tests were made. Quantities of 300 ml each of double-distilled water and 10 percent H_2SO_4 solution were saturated with nitrogen, using a fritted glass bubbler. Nitric oxide was then bubbled through the N_2-saturated solutions for 30 minutes. The pH of the water was measured after nitrogen saturation and again after the nitric oxide treatment with a Beckman pH meter, Model G. Spot tests for nitrate and nitrite ions were made on the N_2-saturated H_2SO_4 solution before and after nitric oxide bubbling. The spot tests were the iodide-starch test for $(NO_2)^-$ and the diphenylamine test for $(NO_3)^-$ as described by Fe.gl.[3]

Additional corrosion tests of the abraded specimens were made in the N_2 and nitric oxide-treated 10 percent H_2SO_4 solutions. Enough urea was added to some of the test solutions to remove all traces of the nitrite ion. Corrosion tests also were made of abraded specimens in 10 percent H_2SO_4 solutions, to which were added increasing amounts of 10 percent nitric acid.

The stainless steel specimens used in the sorption tests consisted of 40 strips, each measuring 0.005 x 1 x 7 inches. The projected area of this specimen was 0.36 square meter. The strips were sheared to size and then carefully sanded to remove all crimped edges. Pickling was accomplished as described above except that the specimens were dried in air before being sealed in the Pyrex sorption apparatus shown schematically in Figure 2. This apparatus was modeled after that of Armbruster and Austin.[4] In essence, it consists of two carefully calibrated volumes, one of which contains the specimen and the other of which is connected to a McLeod gauge. In this apparatus the specimens were exposed to an atmosphere of oxygen or nitric oxide. The gas sorbed on the metal surface caused the pressure to fall, and the quantity sorbed was calculated from pressure drop. Gas pressures of up to 15 mm can be handled in the apparatus.

McLeod gage readings were made with a cathetometer and could be reproduced to within two percent.

Before the start of each sorption test, the stainless steel specimen was outgassed at room temperature at pressures of 5 x 10^{-6} mm Hg or less. The true surface area of specimens treated in this manner was determined using the Brunauer-Emmett-Teller method. The adsorption of carbon monoxide, produced by the reaction of formic acid with warm concentrated sulfuric acid, was measured at —183 C.

Figure 3 is a schematic diagram of a special apparatus used for corrosion tests of the Type 302 stainless steel in N_2-saturated 10 percent H_2SO_4 solution. Abraded specimens were prepared as described above, dried in air, then placed in the specimen chamber which, in turn, was connected to the apparatus by a standard taper joint. The entire apparatus was evacuated to 10^{-6} mm Hg or less, then nitric oxide, at a pressure of one atmosphere, was admitted to the specimen chamber. After overnight exposure, the nitric oxide was pumped out and N_2-saturated 10 percent H_2SO_4 solution at 50 C was admitted to the specimen chamber. This apparatus was modeled after that of Fontana and Beck.[5]

Results and Discussion

Figure 4 shows the time-potential curves for abraded and pickled specimens of Type 302 stainless steel in oxygen-saturated 10 percent H_2SO_4, and for similar specimens in nitric oxide-saturated sulfuric acid solution. The pickled and abraded specimens exposed to the nitric oxide atmosphere and nitric oxide-saturated 10 percent sulfuric acid showed very high positive potentials and no visible corrosion occurred. The specimens were not tarnished and the sulfuric acid solutions were not discolored. The pickled specimen exposed to the oxygen atmosphere and oxygen bearing acid was also passive though its potential was not so noble as that of the specimens exposed to nitric oxide-treated acid. On the other hand, abraded speci-

mens exposed to oxygen-treated 10 percent sulfuric acid solutions were visibly blackened in every case. A tendency for the potential to become more noble with increasing time of exposure was observed. However, violent hydrogen evolution continued after two hours. Duplicate tests were made for every set of conditions.

Table 1 shows the results of corrosion tests of abraded specimens of Type 302 stainless steel in 10 percent H_2SO_4 solution at 50 C. Five ml of 10 percent HNO_3 in 300 ml of 10 percent H_2SO_4 (or bubbling the solution with nitric oxide for 30 minutes) renders the specimen passive. This amounts to one part HNO_3 to 60 of H_2SO_4.

Test 6 (300 ml 10 percent H_2SO_4 plus 45 ml 10 percent HNO_3 plus 10 grams urea) shows that HNO_3 without the presence of HNO_2 is not nearly so effective a passivator. This solution gave a positive test for nitrite ion before the addition of urea. Afterwards, the test was negative and the stainless specimen immersed in this solution corroded noticeably. Forty-five ml of 10 percent HNO_3 are at least nine times the amount necessary to passivate as shown by Test 2. A mild boiling action accompanies the addition of urea since N_2 and CO_2 are products of the reaction of urea with HNO_2.

Nitric oxide apparently reacts with water to a considerable extent, as the pH of nitrogen-saturated double-distilled water decreases from 6.5 to 2.3 after 30 minutes exposure to bubbling nitric oxide at room temperature. Neither HNO_2 nor HNO_3 could be detected in the water before nitric oxide exposure, but positive results were obtained afterward.

Spot tests of 10 percent H_2SO_4 solutions before and after nitric oxide exposure gave similar results—the presence of HNO_2 and HNO_3 could be detected after the solution was bubbled with nitric oxide for 30 minutes, but not before. It should be mentioned that the iodide-starch test for nitrous acid is very positive, while the diphenylamine test for nitric acid after removal of HNO_2 by urea is relatively weak. Conversely, solutions of 10 percent H_2SO_4 to which one part in 60 of 10 percent HNO_3 has been added give a very strong test for HNO_3 and a relatively weak test for HNO_2. Both solutions passivate stainless steel.

Tests 7 and 8 in Table 1 show that the removal of HNO_2 from nitric oxide-exposed 10 percent H_2SO_4 destroys the passivating effect. The addition of 10 grams urea to 300 ml of the nitric oxide-exposed H_2SO_4 solution was accompanied by vigorous boiling as the HNO_2 was destroyed.

The sorption tests were performed on pickled Type 302 stainless steel specimens having a projected area of 0.36 square meter. A roughness factor of four was measured for the stainless steel specimens by the adsorption of carbon monoxide at —183 C. Thus, each specimen had a true surface area of about 1.4 square meters. This method of determining the true surface area of stainless steel specimens was used successfully by Armbruster.[6]

Figure 5 shows the sorption of oxygen at room temperature on an outgassed pickled stainless steel surface. There appears to be rapid initial sorption followed by a slower pick up of oxygen at a constant rate. The capacity of the

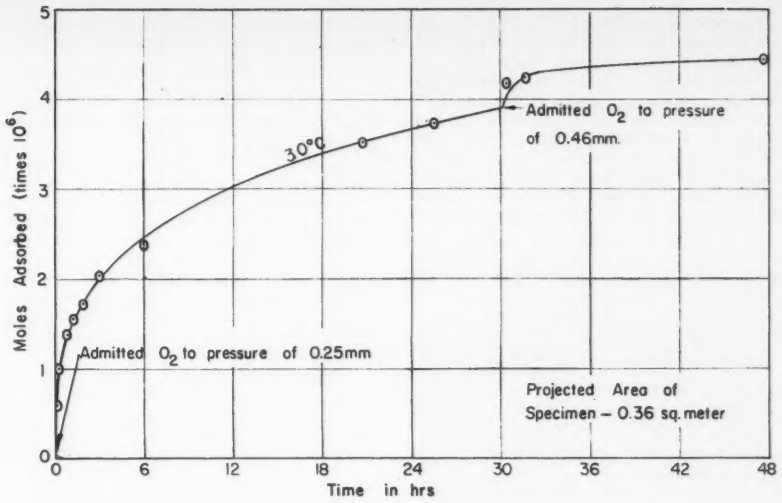
Moles Adsorbed (times 10^6)
Time in hrs
30°C
Admitted O_2 to pressure of 0.46mm.
Admitted O_2 to pressure of 0.25mm
Projected Area of Specimen – 0.36 sq. meter

Figure 5—Sorption of O_2 at 0 C and 30 C on outgassed pickled stainless steel.

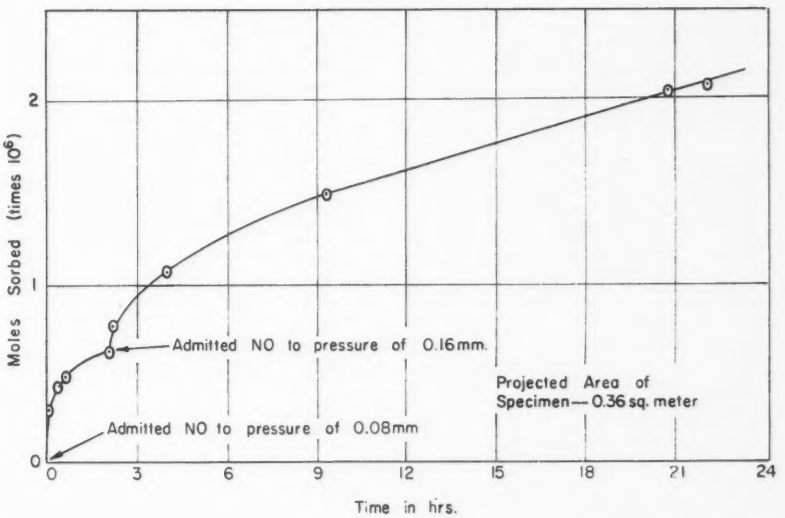
Moles Sorbed (times 10^6)
Time in hrs.
Admitted NO to pressure of 0.16mm.
Admitted NO to pressure of 0.08mm
Projected Area of Specimen— 0.36 sq. meter

Figure 6—Sorption of NO at 30 C on outgassed pickled stainless steel.

pickled stainless steel surface to take on more oxygen is not limitless. After about 48 hours of continuous exposure to oxygen at the pressures shown on Figure 5, the surface appeared to be saturated and no further pressure drop was detected. Increasing the pressure of oxygen to 10 mm produced no further sorption. Assuming that a molecule of oxygen occupies 14.1 square angstroms, 5×10^{-6} moles of oxygen would cover about 30 percent of the true surface. There is no evidence to indicate that the adsorption is molecular, however. This oxygen is held on the stainless steel surface irreversibly.

Figure 6 is a time-sorption curve for nitric oxide on pickled outgassed stainless steel at room temperature. The shape of this curve is similar to that of the oxygen curve discussed above. The mechanism by which nitric oxide is picked up by a stainless steel surface at room temperature would appear to be related to that of oxygen pickup. The stainless steel surfaces appear to become saturated with nitric oxide before as much as a monolayer is adsorbed. Again, the sorption is irreversible and there is no evidence indicating that the gas is molecularly adsorbed.

McClellan and Hackerman[7] measured the sorption of nitric oxide and O_2 on 18-8 powder at room temperature. They reported 1.4 monolayers of oxygen and 2.9 layers of nitric oxide on such powder after it had been baked in vacuum. It is not surprising that such a large difference exists between these data and those reported above, the methods of surface preparation being so radically different. The stainless steel powders used by McClellan and Hackerman were prepared by corrosion of sensitized stainless steel while the strips used in the present experiments were pickled, washed, and

dried in air before being sealed in the adsorption apparatus. It is likely that the strips already had a film of irreversibly held oxygen before being submitted to the vacuum. It also is possible that baking in vacuum changes the nature of the surface. At any rate, the present sorption tests show that oxygen and nitric oxide act similarly on pickled stainless steel surfaces, which previously have been exposed to air, with more oxygen than nitric oxide being sorbed.

It is difficult to see how such a small amount of nitric oxide on a stainless steel surface could cause passivity. In order to find out if adsorbed nitric oxide is capable of producing passivation of an abraded specimen, tests were made in the apparatus shown in Figure 3. Abraded specimens exposed to nitric oxide at one atmosphere pressure for 12 hours and then immersed in N_2-saturated 10 percent H_2SO_4 corroded rapidly.

Since the nitric oxide sorbed on the stainless steel surface is irreversibly held, pumping out the chamber before admission of the sterile acid should not alter the surface.

Conclusion

It has been found that nitric oxide causes passivation of abraded Type 302 stainless steel surfaces in 10 percent H_2SO_4 solution. This passivation apparently is due to the presence of HNO_2 in the acid. Corrosion tests of nitric oxide-exposed specimens in sterile H_2SO_4 solutions show that nitric oxide adsorption alone is not responsible. Removal of HNO_2 from the solutions by the addition of urea destroys the passivating effect of nitric oxide-treated H_2SO_4 solutions.

The addition of one part of 10 percent HNO_3 to 60 parts of 10 percent H_2SO_4 has a similar passivating effect. This effect is destroyed also by the addition of urea. Thus, it follows that neither nitric oxide nor HNO_3 alone causes the passivity. The presence of HNO_2 is required.

Acknowledgment

Portions of this work are based on L. F. Lockwood's Masters thesis, June, 1955, and W. P. McKinnell's PhD thesis, December, 1956, at the Ohio State University.

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Any discussions of this article not published above will appear in the June, 1958 issue

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A Kinetic Study of Acid Corrosion of Cadmium*

By HENRY WEAVER, JR. and CECIL C. LYNCH

Abstract

The kinetics of the reaction of dilute hydrochloric acid with cadmium metal, molded, electroplated on brass, and dipped on brass, have been investigated by means of calibrated conductance measurements. The effect of annealing molded cadmium samples was to reduce the corrosion rate. Changing the speed of spinning the cylindrical sample increased the rate with increase in rotational velocity, up to a constant value at high velocities. The reaction rate at three acid concentrations and at three temperatures were determined. The activation energy is small, between one and two kilocalories per mole of cadmium reacting. In all cases the reaction was zero order over 90 percent of the reaction process. The rate controlling step has been estimated as that of diffusion through the layer surrounding the cadmium surface. 3.8.4

Introduction

THE WORK REPORTED here differs from previous studies of corrosion of cadmium by hydrochloric acid in that the process was studied in dilute solutions of the acid and no depolarizers were used to remove the hydrogen which formed at the surface of the cadmium. Jablczynski and Wajchselfisz[1] studied the rate of solution of cadmium in hydrochloric acid as early as 1929. They used 5 normal acid, however, and apparently induced a passive state on the cadmium since the reaction slowed quite rapidly. King and others[2,3,4,5] in later studies used depolarizers to remove the hydrogen film that develops about the cadmium. Other studies have investigated the effects of additional chloride ions on the solution.[6,7]

This investigation was aimed at measuring the kinetic rate of attack of dilute aqueous hydrochloric acid on cadmium metal under varying conditions through the use of a carefully controlled conductance technique. This required separate calibration curves of concentration of the cadmium ion vs specific conductance of the solutions for each acid concentration and each temperature employed. Figure 1 shows a typical calibration curve. The method proved quite satisfactory, since measurements could be made rapidly with no part of the sample lost during analysis.

Experimental

A modified Jones conductivity cell was used. The filling stem of the cell was made to turn downward instead of upward. This was then allowed to dip into a three-necked flask, in which the determination was carried. A 29/42 standard taper ground glass connection was made between a male fitting fastened to the cell arm and the flask. The cadmium sample was placed in the flask through the center neck of the flask, and the third neck was used as a vent for the hydrogen gas produced in the reaction. Two rubber stoppers with a film of stopcock grease between them served as a gas tight seal on the stirrer shaft that held the sample. A sketch of the apparatus is given in Figure 2.

★ Submitted for publication February 25, 1957.

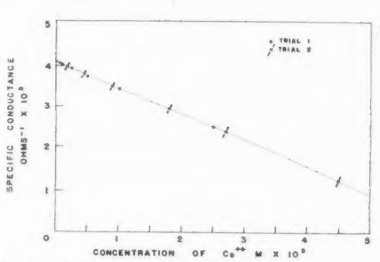

Figure 1—Calibration with 0.010 m HCl at 25.0 C.

Measurements of the conductivity were made with a Wheatstone bridge using a thousand cycle voltage source. The null point was detected with headphones.

The reaction vessel and conductivity cell were submerged in a water bath which maintained the temperature within ∓0.02 degrees C.

Standard solutions for calibration plots of cadmium ion concentration against conductance were made by dissolving known weights of cadmium metal in hydrochloric acid of the concentrations used, and then making volumetric dilutions with acid of the same strength.

The cadmium samples used were in the shape of cylinders with a diameter of 1.0 to 1.5 centimeters. They were tapped at one end and fastened to a 3/16 inch shaft. Part of the sample was covered with plastic Korolac in order to expose a known surface area. Preliminary tests were made on this plastic film to show that diffusion of the acid through it was negligible during the time required for any one experiment.

Weaver

Lynch

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For annealing, samples were placed in a nitrogen atmosphere in an oven at 295 to 300 C and the temperature slowly lowered to room temperature over a period of three days. Dipped samples were prepared by dipping tapped brass cylinders of the same size in molten cadmium at 450 C. Electroplating of cadmium was done on brass according to the method of Weisberg.[8]

TABLE 1—Summary of Rate Constants for Various Samples

Run No.	Type of Sample	Stirring Speed, RPM	Temp Degrees C	Conc of HCl – m	Rate Constant (m/sec x cm²) X 10⁹
1	Unannealed	380	25.0	0.010	3.3
2	Unannealed	380	25.0	0.010	3.7
3	Unannealed	380	25.0	0.010	4.2
4	Unannealed	380	25.0	0.010	4.2
5-9	Annealed	380	25.0	0.010	3.8
19,20	Annealed and Gassed	1200	25.0	0.010	10.7
21,22	Annealed	1200	15.0	0.0050	9.8
23,24	Annealed	1200	15.0	0.010	10.8
25,26	Annealed	1200	15.0	0.020	14.0
27,28	Annealed	1200	25.0	0.0050	10.2
15,17	Annealed	1200	25.0	0.010	11.3
29,30	Annealed	1200	25.0	0.020	14.5
31,32	Annealed	1200	35.0	0.0050	13.0
33,34	Annealed	1200	35.0	0.010	14.4
35,36	Annealed	1200	35.0	0.020	15.0
37-39	Electro-plated	1200	25.0	0.010	20.8
40	Dipped	1200	25.0	0.010	22.7
41,42	Dipped	1200	25.0	0.010	18.8
43	Dipped	1200	25.0	0.010	16.2

TABLE 2—Effect of Stirring Speed on Rate Constant

Run No.	Stirring Speed, RPM	Rate Constant (m/sec x cm²) x 10⁹
5-9	380	3.8
10	600	6.1
11	700	6.8
12	800	8.2
13	800	10.0
14	900	9.6
15,16	1000	10.3
17	1200	10.1
18	1400	9.7

Note: All runs reported were with annealed samples of cadmium in 0.010 HCl at 25.0 C.

TABLE 3—Tabulation of Logs of Rate Constants and Reciprocal of Absolute Temperature for Annealed Samples of Cadmium

Conc of HCl – m	Temp, Degrees Kelvin	1/T x 10³	Rate Constant (m/sec x cm²) x 10⁹	Log of Rate Constant
0.0050	288	3.472	9.8	0.991
0.0050	298	3.356	10.2	1.009
0.0050	308	3.245	13.0	1.113
0.010	288	3.472	10.8	1.033
0.010	298	3.356	11.3	1.053
0.010	308	3.245	14.4	1.158
0.020	288	3.472	14.0	1.146
0.020	298	3.356	14.5	1.161
0.020	308	3.245	15.5	1.190

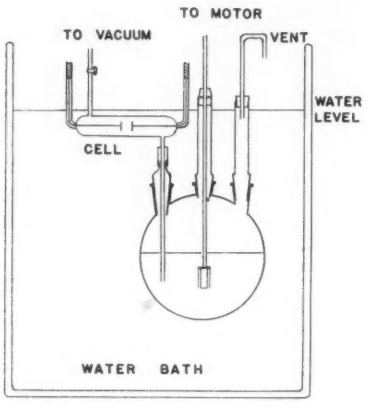

Figure 2—Apparatus for kinetics measurements.

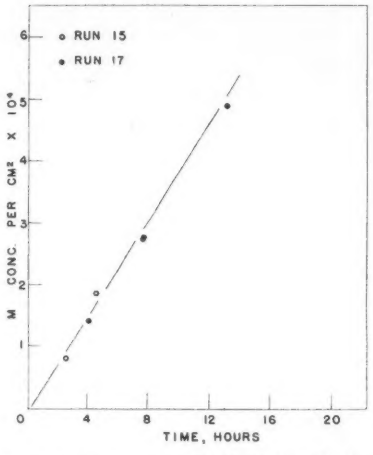

Figure 3—Cadmium in 0.010 m HCl at 25.0 C.

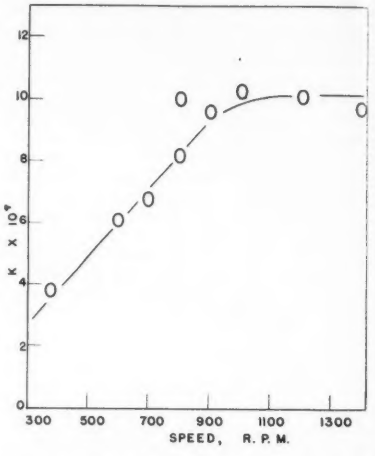

Figure 4—The effect of stirrer speed on the rate constant. K is molality of Cd^{++} per cm^2 per sec. Cadmium is in 0.010 m HCl at 25.0 C.

Results and Discussion

In every run the kinetics were examined by measuring the changing conductance with time. On plotting the concentration of the cadmium ion vs time, straight lines resulted for the reaction up to 90 percent completion, indicating zero order for the process of acid corrosion of cadmium. Figure 3 shows a typical plot.

The effects of annealing the sample, gassing the sample with nitrogen, electroplating and dipping are shown in Table 1. A comparison of the first four runs which were made on unannealed molded cadmium with the next several runs shows that the unannealed samples had much more variation in the value of the rate constant than did the annealed samples. The conventional interpretation of strains in the metal making it less uniform and more reactive seems applicable here.

Since no appreciable change is noted in the rate constants of the runs made with samples that were gassed with nitrogen to remove oxygen and those not, it can be assumed that oxygen does not enter into the mechanism of the reaction.

Both the electroplated and dipped samples showed much higher rate constants than the annealed samples. This is readily explained by assuming considerable strain in the thin coatings.

The effect of variation in the speed of stirring on cadmium in 0.010 m hydrochloric acid at 25.0 C is shown in Table 2 and Figure 4.

A calculation of the logs of rate constants and reciprocal of temperature, taken from Table 1, is shown in Table 3 and plotted in Figure 5. From the slope of the lines in this plot the activation energy of the overall process can be calculated. For 0.0050 m and 0.010 m acid it is 2 ∓ 0.3 K cal per mole. For 0.020 m acid it is 1 ∓ 0.5 K cal per mole. It can be concluded therefore that within the experimental error the activation energy has a value of from 1 to 2 K cal per mole.

Since the reaction studied is of zero-order, the question of what is the rate controlling step should be considered. It is obvious that the diffusion of the ions of hydrogen through the solution is a factor, but that does not change the order of the reaction. Diffusion through the layer of solution with a diffusion gradient is probably the rate controlling step. Further work is anticipated to use this method of analysis to investigate the effect of variation of chloride ions concentrations.

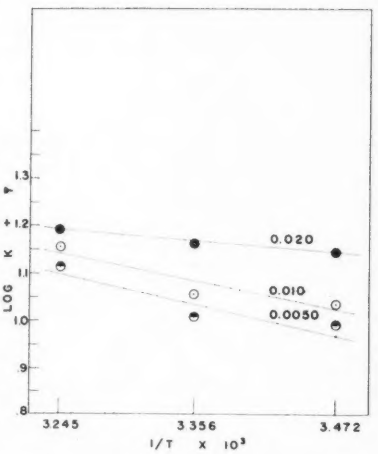

Figure 5—Plot of logs of rate constants against the reciprocal of absolute temperature for three concentrations of hydrochloric acid.

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Any discussions of this article not published above will appear in the June, 1958 issue

Publication 58-2

NACE TECHNICAL COMMITTEE REPORT

Compilation and Correlation Of High Temperature Catalytic Reformer Corrosion Data*

A Contribution to the Work of NACE Technical Group Committee T-8*

By G. SORELL

Introduction

THE RECENT phenomenal growth of catalytic reforming into a major oil refining process for producing high octane motor fuels has generated widespread concern with respect to the serious corrosion problem frequently encountered in this type process. A recently published survey report presented to the American Petroleum Institute on "Corrosion in Catalytic Reforming and Associated Processes"[1] indicates that the majority of 31 companies interviewed had experienced excessive metal loss accompanied by serious operational difficulties. The most common of these difficulties involved plugging of processing equipment by loose corrosion products, predominantly metal sulfide.[1] The corrosive agent responsible was identified conclusively as hydrogen sulfide in the hot hydrogen rich gas mixtures common to catalytic reforming units.

In order to combat this dual problem, a number of petroleum refining companies independently embarked on corrosion investigations and test programs. A number of valuable papers based on these studies have been published since the early part of 1956. Although the reported work was largely limited to an empirical approach in the form of laboratory, pilot plant and refinery corrosion rate measurements, some fundamental studies also have been carried on. Among the latter, an outstanding contribution is the thermodynamic analysis presented by the Standard Oil Company of Indiana.[2, 22]

In an effort to explore the problem further, two industrial "Study Committees" were formed (API Panel on Reformer Corrosion and NACE Task Group T-5B-2),[2] with participating membership drawn from practically all major American petroleum refining companies. As a result of this unre-

About the Author

G. SORELL is materials and corrosion engineer with the M. W. Kellogg Company, New York City, where his duties include specifying materials and corrosion protection for a large variety of process plants in the petroleum refining and chemical processing fields. He also has worked in his company's process development and design engineering departments. Born in Vienna, Austria, he came to this country in 1939 and served with the U. S. Army in Europe. Mr. Sorell holds BS and MS degrees in chemical engineering, both from Princeton University. He is a member of the National Association of Corrosion Engineers, the Scientific Research Society of America, Sigma Xi and Phi Beta Kappa.

★ A paper presented at a meeting of NACE Technical Group Committee T-8, Oklahoma City, Oklahoma, October 1, 1957.

* M. E. Holmberg, chairman.

(1) Although large accumulations of metallic oxides are frequently experienced in catalytic reforming units practicing "in situ" regeneration of catalyst, they result from oxidation of sulfide scale formed during reaction, not from direct attack of the metal by the oxidizing regeneration atmosphere.

(2) NACE Task Group T-5B-2 (Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry) has been absorbed in the newly formed NACE Technical Committee T-8 (Corrosion of Petroleum Refinery Equipment).

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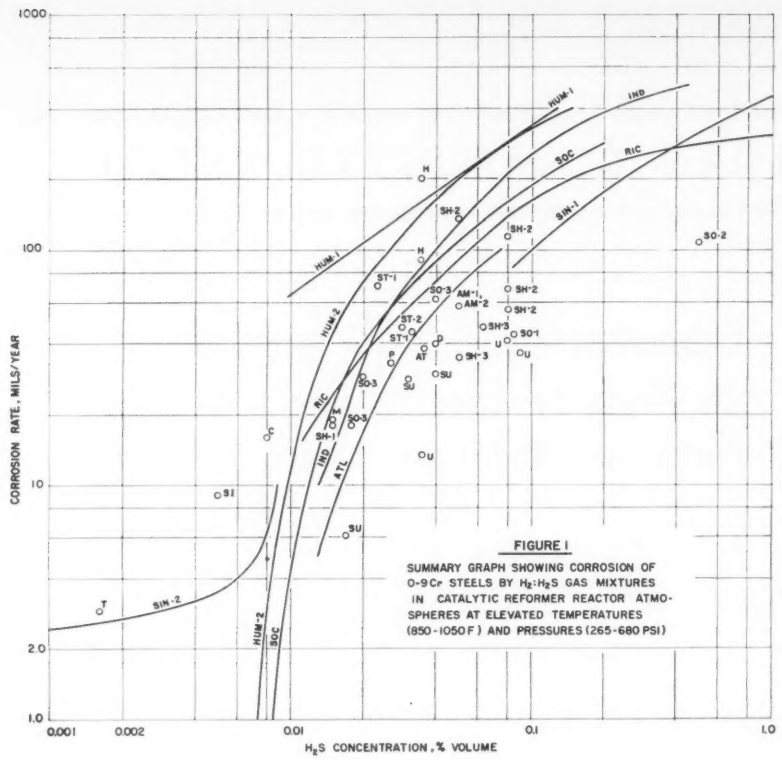

FIGURE 1
SUMMARY GRAPH SHOWING CORROSION OF 0-9 Cr STEELS BY H_2:H_2S GAS MIXTURES IN CATALYTIC REFORMER REACTOR ATMOSPHERES AT ELEVATED TEMPERATURES (850-1050 F) AND PRESSURES (265-680 PSI)

Figure 1—Summary graph showing corrosion of 0-9 Cr steels by H_2:H_2S gas mixtures in catalytic reformer reactor atmospheres at elevated temperatures (850-1050 F) and pressures (265-680 psi).

Abstract

In this survey report on high temperature catalytic reformer corrosion, published and privately transmitted corrosion data are compiled and summarized. The data, contributed by 15 petroleum refining companies, comprise results obtained from laboratory, pilot plant, and commercial plant corrosion studies, as well as inspection measurements on operating equipment. Correlations directly applicable to equipment design are presented.

The corrosion is attributed to small quantities of hydrogen sulfide present in the hot hydrogen rich gas mixtures. The principal variables affecting the rate of attack are temperature and hydrogen sulfide concentration. Other factors such as pressure, hydrogen content, time of exposure, cycling conditions and scale properties also are discussed in relation to their effect on corrosion.

The data show conclusively that carbon and chrome-moly steels are equally susceptible to sulfide attack. The intermediate straight chrome steels (11 percent to 16 percent Cr) show a moderate but generally unreliable degree of improvement. The austenitic chromium-nickel stainless steels have good resistance. Aluminum coatings applied by dipping, spraying or diffusion methods exhibit outstanding corrosion resisting properties, provided the coating is continuous and remains firmly bonded to the steel.

The paper concludes with a general review of catalytic reformer corrosion problems, including a brief discussion of corrosion mitigation by hydrogen sulfide removal (amine treating) or catalytic desulfurization of the feed. 8.4.3

stricted pooling of knowledge and free exchange of experience, excellent progress has been made toward a sound understanding of, and the development of economic solutions to the corrosion problem under consideration. In view of the highly competitive situation existing among the different proprietary catalytic reforming processes, the industry is to be congratulated on their foresight and wisdom in launching these joint efforts.

Objective and Scope of Paper

The primary objective of this paper is to compile and summarize in a single convenient reference the available quantitative high temperature corrosion data pertinent to catalytic reforming processes, and to correlate the findings in a form applicable to process equipment design. Toward this end, corrosion rate measurements obtained from the published literature and from heretofore unpublished reports are presented in tables and graphs, and are correlated in the form of three summary graphs (Figures 1, 2 and 3) and one correlation graph (Figure 4).

The data originate from a wide variety of sources ranging from laboratory investigations to actual operating experience by 15 contributing petroleum refining companies. Including the work performed in connection with five extensive laboratory studies,[2,4,5,6,7] the total number of individual measurements represented in this compilation is estimated to be well above one thousand.

The field corrosion data herein presented have been obtained exclusively from catalytic reforming plants and therefore do not reflect the more corrosive conditions typical of catalytic desulfurization units. A companion paper summarizing catalytic desulfurizing corrosion is in the early stages of preparation by the author and will be completed when more service experience on these process units becomes available.

Organization and Presentation of Data

In Tables 2 to 27 and Figures 5 to 15, the high temperature corrosion rates measured or estimated by the individual investigators are compiled alphabetically, according to the contributors' company affiliations. The type of data presented falls into three broad categories.

1. Laboratory and pilot plant corrosion investigations.
2. Corrosion coupon tests in commercial units.
3. Inspection of operating equipment.

For ready reference, a single summary chart (Table 1) has been prepared, showing corrosion rates and the corresponding ranges of environmental conditions for all the data presented in the individual summaries. This chart also serves as master chart for use in conjunction with the summary graphs (Figures 1, 2 and 3). These summary graphs present the collected data in a correlated form which permits rapid visual comparison of the individual findings. Three separate summary graphs were chosen in preference to a single plot for sake of clarity; they are respectively Figure 1 for carbon and chrome-moly steels (0 to 9 percent Cr), Figure 2 for intermediate chrome steels (11 percent to 16 percent Cr) and Figure 3 for stainless steels (18 percent Cr/8 percent Ni). (Note that the corrosion rate axis of Figure 3 is shifted by a factor of 10 relative to the scales used in Figures 1 and 2.)

In the correlation graph (Figure 4), the compiled data are condensed into a simple unified plot in which the individual data points and curves are supplanted by "bands" of corrosion rates for the three classes of steel. This form of presentation offers a rapid and convenient means for predicting the range of anticipated corrosion rates for design purposes.

Discussion of Corrosion Data

The corrosion of carbon and alloy steels widely experienced in the high temperature sections of catalytic reforming units has been conclusively identified as a form of sulfide attack, and is attributed to the presence of relatively small quantities of hydrogen sulfide in the hot reducing gas mixtures common to this type of unit. The magnitude of this attack, illustrated by the data in the three summary graphs (Figures 1, 2 and 3), gives clear evidence of an exceptionally severe form of sulfide corrosion which is without previous parallel in petroleum refining experience.

Since this survey paper is intended primarily to present and reorganize a collection of empirical data for practical application, an analytical discussion of the physico-chemical or metallurgical aspects of the corrosion phenomenon appears inappropriate. The discussion material is therefore restricted to a review of factors which are considered of direct benefit to the engineer for proper interpretation, evaluation and utilization of the data. These factors are the specific effects of alloying additions and environmental variables on corrosion rate, as well as the general validity of the measurements.

Alloy Content of Steel

The accumulated data show almost without exception that the chrome-moly steels (up to about 9 percent Cr) offer no improved resistance to attack over plain carbon steel; there is some evidence that the popular 5 percent Cr-½

TABLE 1—Summary Chart of Corrosion Measurements and Corresponding Environmental Conditions

SOURCE (Company)	Symbols in Figures 1, 2 and 3	Reference	Type Data*	Temperature (Deg. F)	Pressure (Psig)	H_2S Conc. (Vol. %)	Duration (Days)	Materials	Corrosion Rate (Mils/Year)
American Oil	AM–1	12	I	950	265–400	0.03–0.07	36–89	1¼ Cr, 2¼ Cr	59
	AM–2	12	C	950–1025	265–400	0.03–0.07	89	0–9 Cr 12 Cr 18 Cr/8 Ni	59 30 13
Atlantic	ATL	7	L P C I	900	500	0.013–0.074 0.011–0.13 0.015–0.27	1–365	0–5 Cr 11½–13½ Cr 18 Cr/8 Ni	5–100 1– 60 1– 8
	AT	12	I	900	500	0.036 0.016	180	2¼ Cr 12 Cr	38 7
D–X Sunray	D	12	I	870	400	0.04	180	5 Cr	40
Canadian Petrofina	C	13	C	890–920	310–330	0.008	90	0–9 Cr 12 Cr 18 Cr/8 Ni	16 8 2.5
Humble	HUM–1	6	C	900–956	300	0.013–0.14 0.013–0.11	1–4	0–12 Cr 18 Cr/8 Ni	73–398 5–42
	HUM–2	6	P	1000	300	0.007–0.15	4	0–12 Cr 18 Cr/8 Ni	1–400 0.4–60
	H	6	I	850–975 900–1025	300	0.035	127	1¼ Cr C. S., 2¼ Cr	90 200
Major U.S. Ref'g. Comp.	U	14	C	875–960	500–535	0.035–0.09	139–577	2¼ Cr, 5 Cr 12 Cr 18 Cr/8 Ni	13.5–41.5 11–27 3.7
Major West Coast Ref'g. Comp.	M	15	C	900–920	540–565	0.015	72	0–5 Cr 12 Cr 18 Cr/8 Ni	18.9 8.3 4.5
Pure Oil	P	12	I	900–1000	500	0.026	240	2 Cr	33
Richfield	RIC	9	L P C I	950	400	0.011–1.0 0.014–1.0 0.025–1.0	16.7	0–9 Cr 12 Cr 18 Cr/8 Ni	15–310 10–240 3.5–20
Shell Oil	SH–1	17	C	950	625	0.015	71	0–9 Cr 12 Cr	18 8
	SH–2	17	I	950 955–1135	680 670	0.05–0.08 0.08	148–758 758	5 Cr 9 Cr	56–135 68
	SH–3	12	C	925	680	0.05–0.08	148–251	0–9 Cr 12 Cr 18 Cr/8 Ni	35–47 15–22 4.3–7.2
Sinclair	SIN–1	4	L	950	500	0.08–1.0 0.1–1.0 0.1–1.0	4.2	0–7 Cr 13 Cr 18 Cr/8 N i	80–450 20–210 3–48
	SIN–2	4	C	910–940	500	0.001–0.0089	212–382	0–7 Cr 13 Cr 18 Cr/8 Ni	2.4–10 1.9–6.5 0.55–0.6
	SI	12	I	900–1025 900–950	500	0.005	120–270	5 Cr 12 Cr	9 6.5
Socony Mobil	SOC	5	L	985	485	0.009–0.2 0.017–0.2 0.06–0.2	6.3–20.8	0–5 Cr 7–16 Cr Cr/Ni	1–285 1–102 1–8.7
	SO–1	5	L	905	460	0.085	21	5 Cr	44
	SO–2	18	C	885–935	175	0.5	55	0–9 Cr 12–16 Cr Cr/Ni	108 21 5
	SO–3	19	C	885–920	550–575	0.018–0.04	50.5–213	0–9 Cr 12 Cr Cr/Ni	18–62 13–23 0.7–4.3
Standard Oil (Ind)	IND	2	L	900	285	0.013–0.44	0.7–11.7	0–9 Cr 12 Cr 18 Cr/8 Ni	10–500 4–200 1.3–62.5
	ST–1	2	C	950 950–1000	300	0.023 0.032	16.5–46	C.S.	70 45
	ST–2	3	C	1030	340	0.029	47	0–9 Cr 18 Cr/8 Ni	37.5 3.3
Sun Oil	SU	14	C	925–960	300–600	0.017–0.040	188–395	0–5 Cr 12 Cr 18 Cr/8 Ni	6.1–29.7 5.8–11.7 0.7–1.9
Texas	T	21	C	940	600	0.0016	133–668	0–5 Cr 12 Cr 18 Cr/8 Ni	2.9 1.9 0.3

* L—Laboratory Corrosion Tests
P—Pilot Plant (Corrosion Tests or Equipment Corrosion)
C—Commercial Unit Corrosion Tests
I—Inspection of Commercial Operating Equipment.

percent Mo composition may even be inferior to unalloyed steel.

The intermediate chrome steels (11 percent to 16 percent Cr), principally the widely used AISI Types 405 and 410 (11½ percent to 13½ percent Cr), show only a moderate degree of improvement over carbon or lower alloy steels, in the order of approximately 50 to 100 percent. Several investigations do not reveal even this small difference in corrosion resistance between carbon and intermediate chrome steels; in support of this, note the identical position of curves HUM-1 and HUM-2 in the summary graphs for 0 to 9 percent Cr and 11 percent to 16 percent Cr steels (Figures 1 and 2 respectively). Generally, the performance of the intermediate chrome steels is subject to greater individual variation than that of the lower alloy steels, as is evident from the larger scatter of data in Figure 2 in contrast to Figure 1.

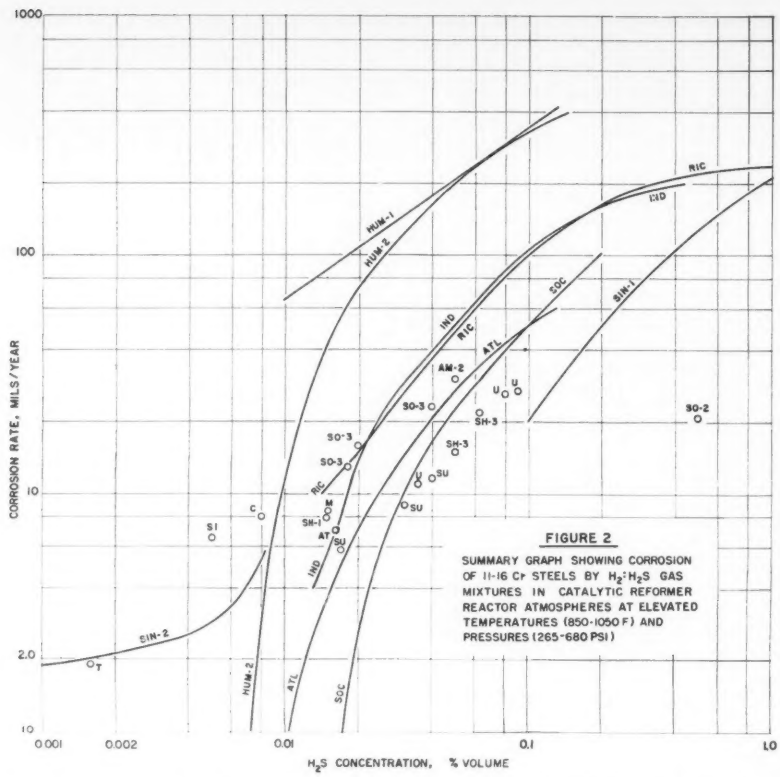

Figure 2—Summary graph showing corrosion of 11-16 Cr steels by H_2-H_2S gas mixtures in catalytic reformer reactor atmospheres at elevated temperatures (850-1050 F) and pressures (265-680 psi).

The 18 percent Cr/8 percent Ni grades of stainless steel such as AISI Types 304, 316 and 347 all possess equally good corrosion resistance, with rates only about one-fifth to one-tenth those exhibited by carbon and the lower alloy materials. The higher grades of austenitic stainless steels such as AISI Types 309 (25 percent Cr/12 percent Ni) and Type 310 (25 percent Cr/20 percent Ni) are equal or superior to the 18 percent Cr/8 percent Ni grades.

Environmental Factors Influencing Corrosion

The two major variables which control the extent of elevated temperature catalytic reformer corrosion of a given steel are temperature and hydrogen sulfide content of the gas mixture in the reactors and associated equipment. In order to permit a simple and consolidated presentation of the data, only one of these, hydrogen sulfide concentration, is chosen as a variable in the summary and correlation graphs (Figures 1, 2, 3 and 4). This is possible without gross distortion of the correlations because the range of operating temperatures in catalytic reformer units is within fairly narrow limits (approximately 850-1050 F).[3]

Other factors influencing corrosion rate operative in this environment are pressure, hydrogen content, length of exposure and cyclic conditions. It is emphasized that the corrosion rates plotted in the summary graphs (Figures 1, 2 and 3) represent actual physical measurements and are not adjusted for variations in these operating conditions. This use of raw data tends to produce scatter in the correlations in addition to that introduced by omitting the temperature parameter. In order, therefore, to assist in interpreting the data and in evaluating the correlations, the directional effects of temperature and the other environmental conditions on corrosion are presented individually.

Effect of Temperature

Temperature is a strong factor in high temperature hydrogen sulfide corrosion as revealed by the pronounced effect on corrosion rates resulting from relatively small temperature changes. Specifically, the corrosion rate approximately doubles for a 100 F rise in temperature. It is apparent, therefore, that the effect of temperature should not be discounted, since even within the restricted range of normal catalytic reformer operating temperatures the corrosion rate may vary by a factor as high as 3:1.

Effect of Pressure and Hydrogen Content

The total pressure and the hydrogen content of the hydrogen sulfide bearing gas mixture have a direct effect on corrosion. Based on an experimental study, the Standard Oil Company of Indiana[2] offers the following quantitative proportionality:

$$R \propto (P^{0.4})\,(C^{0.4})$$

Where R = Corrosion rate at a given H_2S concentration
P = Total system pressure
C = Sum of volume fractions of H_2S and H_2

The hydrogen sulfide content in catalytic reformer atmospheres is negligibly small compared to the hydrogen content, and consequently C in the above relationship is essentially equal to the volume fraction of hydrogen. Since both P and C are raised to the same fractional power, it follows that an equal percentage change in either pressure or hydrogen content can be expected to cause an identical change in corrosion rate. It should be noted, however, that between the different catalytic reforming processes operating pressures are subject to considerably greater variation (250-750 psi) than hydrogen concentrations (60-90 mol percent). Hence, failure to adjust individual corrosion measurements for pressure tends to produce greater scatter of data points than neglecting differences in hydrogen concentration. Translated into numerical terms, pressure may influence corrosion rate by as much as 55 percent[4] whereas hydrogen can have a maximum effect of only 7½ percent.[5]

Effect of Exposure Time

The accumulated evidence from laboratory studies indicates that initially corrosion rates are very high but that they drop off rapidly with increasing length of exposure. The experiments conducted by Backensto et al (Reference 5, Figure 4) show a sharp continuous drop in corrosion rate during the first 100 hours; in the remaining 700 hours of exposure, only small further decrease of rate takes place. The relatively short experimental runs of laboratory corrosion studies usually are reflected by corrosion rates appreciably higher than those obtained from prolonged field studies. This trend is clearly borne out in the three summary graphs (Figures 1, 2 and 3), notably by Curves HUM-1 and HUM-2 which are based on 24 to 96 hour periods.

Laboratory studies of the effect of time on corrosion are of limited practical value to the designer and operator of catalytic reforming equipment. Even if carried on for extended periods, these studies cannot properly evaluate the role of the sulfide corrosion scale in stifling further corrosion, since the physical properties and distribution of this scale are largely dependent on the operating histories in a given unit. To obtain a realistic appraisal of the time effect on corrosion rate, one must turn to long time data gathered in operating equipment. A survey of the reported experience reveals some conflicting evidence. The measurements in Tables 9 and 10, for example, indicate no significant differences in corrosion rate for exposures ranging from 139 to 577 days. On the other hand, the figures in Table 16 do show a definite long range trend of decreasing corrosion rates.

The variable effect of exposure time on high temperature hydrogen sulfide attack can be explained by differences in the physical properties of the sulfide corrosion scale relating to its protective value. The majority of reported service experience shows this scale to be of a flaky, permeable and generally nonadherent nature and as such ineffective in blocking or retarding further corrosion. In the relatively few instances where a marked reduction of corrosion rate with time has been reported, the

[3] Metal temperatures in furnaces may range up to 1200 F.

[4] $\left(\frac{750\text{ psi}}{250\text{ psi}}\right)^{0.4} = 1.55$

[5] $\left(\frac{90\text{ mol \%}}{75\text{ mol \%}}\right)^{0.4} = 1.075$

scale was invariably found to be dense and intact. Such denser scale deposits generally are more prone to form on the inside of piping or tubing rather than on flat or convex surfaces because the scale tends to remain under compression. Another factor which can contribute to a tougher and less porous scale is the binding action of small amounts of coke or gum which may be present under certain conditions.

Effecting of Cyclic and Fluctuating Conditions

Cyclic process conditions are considered to have an accelerating effect on corrosion, but quantitative evaluation is difficult. The two cyclic conditions encountered in catalytic reforming are intermittent heating and cooling resulting from shut downs, and exposure to alternating reducing and oxidizing atmospheres, applicable to units practicing "in situ" catalyst regeneration. Both of these conditions tend to loosen and deteriorate the sulfide scale and as such further contribute to the scale's general ineffectiveness as a corrosion barrier.

In contrast to the extreme changes in environment associated with these cyclic conditions, the normal fluctuations in process and operating variables during refinery runs have relatively little effect on corrosion. It is important, however, that appropriately weighted average values of these operating variables be employed in interpreting the corrosion rate measurements.

Accuracy and Validity of Data

In discussing the sources of error to which the data are subject, it is important to recognize and distinguish between two basically different types of possible error. The more obvious one pertains to the accuracy of physical measurements of metal losses and environmental conditions. The other, and more elusive, error has to do with the validity of the measurements which, no matter how accurate physically, may be of limited significance in that they may be valid only for specific conditions in time and space. With reference to the corrosion data in this survey, the following remarks apply primarily to field corrosion studies and inspection results since laboratory experiments usually are carefully planned and controlled, and hence can be expected to give results which are both accurate and reliable.

In general, the accuracy of the data presented in this report is considered to be high and certainly adequate for the intended purpose. Metal loss determinations are reliable providing bench marks are available; with respect to environmental conditions, standard refinery measuring and recording instrumentation are sufficiently accurate. The notable exception arises in connection with the concentration of hydrogen sulfide, which, in the minute quantities frequently present, requires special equipment and skills for accurate detection. The problem is further complicated by the lack of standardization of analytical procedures currently in use, comprising the Tutweiler method, mass spectrometry, MSA Gas Testers and gas absorption followed by potentiometric titration.

The validity and significance of the data is affected by a number of different factors. One of the most serious sources of error derives from calculating corrosion rates in operating equipment

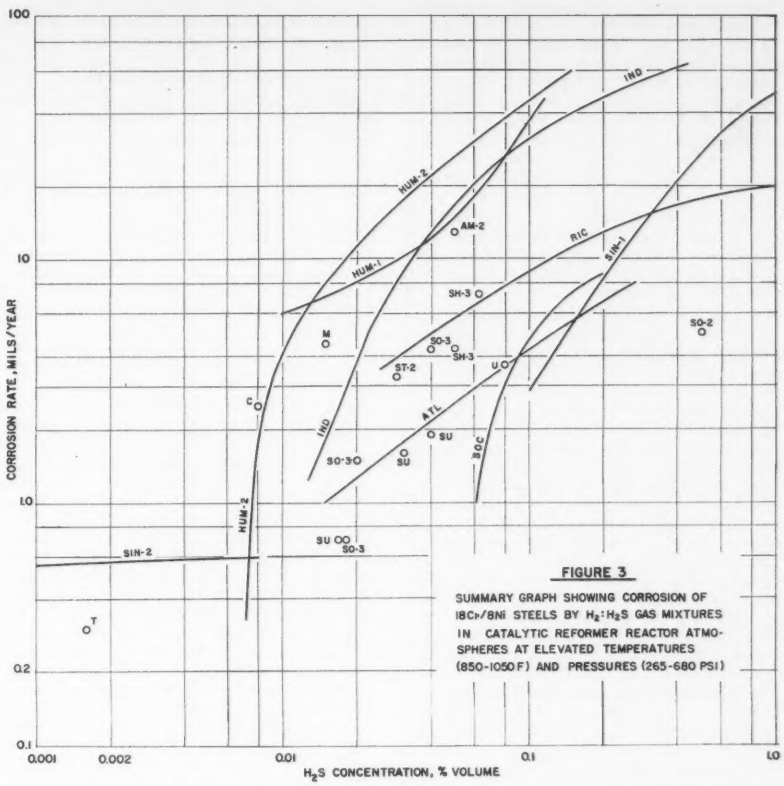

Figure 3—Summary graph showing corrosion of 18 Cr/8 Ni steels by H_2:H_2S gas mixtures in catalytic reformer reactor atmospheres at elevated temperatures (850-1050 F) and pressures (265-680 psi).

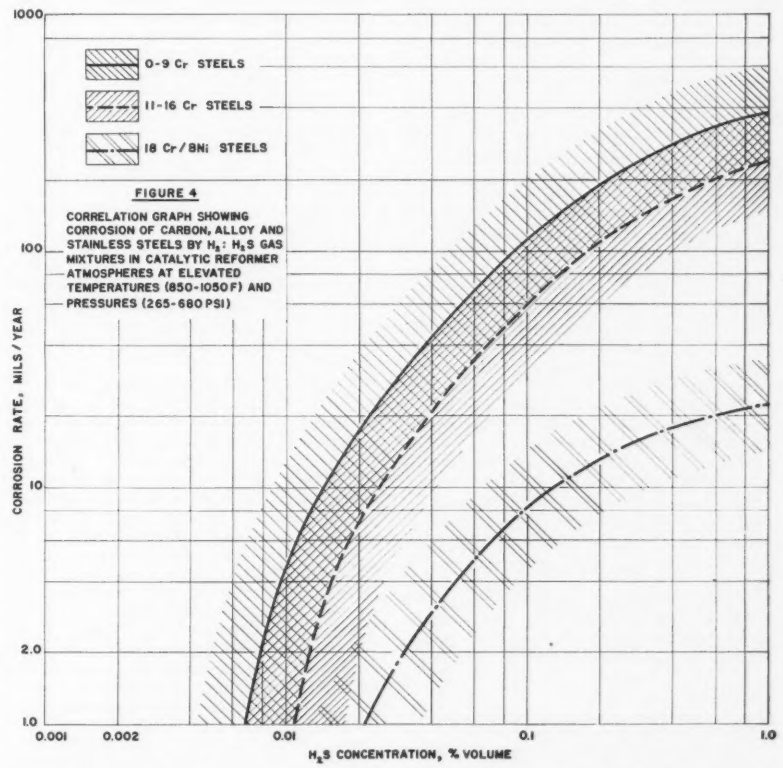

Figure 4—Correlation graph showing corrosion of carbon, alloy and stainless steels by H_2:H_2S gas mixtures in catalytic reformer atmospheres at elevated temperatures (850-1050 F) and pressures (265-680 psi).

from assumed rather than previously measured thickness of corroded parts. This is clearly a dangerous practice since it imposes the use of minimum or average original thicknesses in accordance with applicable ASTM specifications. Corrosion rates thus calculated are subject to gross error because these specifications generally permit wide tolerance latitudes. Estimates of corrosion losses calculated from observed scale thickness in conjunction with an assumed scale factor[6] also may be unreliable because they presuppose that the scale had remained intact during the entire operating run.

A factor which may have an important bearing on the significance of plant corrosion measurements is indeterminate metal loss sustained between operating runs. A recent contribution to the published literature[5] reveals that inhibited acid used for furnace tube descaling can cause considerable corrosion. In one acidizing run, the measured decrease in wall thickness of 2¼ percent Cr-1 percent Mo furnace tubes was equivalent to a corrosion rate of 7 mils per year. Since hydrogen sulfide corrosion in that plant amounted to only 12 mils per year, a vastly exaggerated indication of sulfide corrosion would have been obtained had rate calculations been made without allowing for acidizing corrosion losses. Fortunately, errors of this magnitude cannot creep into the corrosion measurements as the result of neglecting corrosion during "in situ" catalyst regeneration; experience indicates that the hot regeneration gases are essentially non-corrosive.

A difficult problem common to most plant corrosion studies consists of establishing properly weighted average values of the operating variables which can be considered as truly representative for the entire operating run and hence corresponding to the measured corrosion rate. In catalytic reforming units, this difficulty arises primarily in connection with hydrogen sulfide concentrations, which may fluctuate over quite wide limits compared to the smaller percentage variation in temperature, pressure or hydrogen content. Care must be taken in interpreting averages, even when properly weighted, because of the complex non-linear relationships existing between corrosion and its major variables, especially in the vicinity of the thermodynamic cutoff of the corrosion reaction (see Figure 14). The problem of determining representative environmental conditions is further complicated by the fact that the corrosion engineer does not always have personal access to the complete log sheets and service histories, and therefore is dependent on data transcribed or abridged by personnel not necessarily familiar with corrosion work.

In conclusion it should be emphasized that the relatively low order of correlation in the summary graphs (Figures 1, 2 and 3) is the additive result of the various sources of error plus the fact that the individual data points are not adjusted for variations in temperature, pressure, hydrogen content and exposure time. In view of these considerations, it would appear that there is remarkably good consistency in the majority of reported experience.

Correlation of Data

In the correlation graph (Figure 4), the corrosion data accumulated in the summary graphs (Figures 1, 2 and 3) appear in a simple correlation in which three bands take the place of individual points and curves. This presentation is considered the most expedient means for condensing the available information into a clear and compact form for engineering usage. Since, in the author's opinion, the data do not lend themselves to a rigorous statistical treatment, the placement of the three "average" lines and the width of the respective bands are largely dependent on personal judgment.

As can be seen from a comparison of the correlation and summary graphs, the average lines for the three materials classes in Figure 4 are situated appreciably lower than lines which would be obtained from mathematical averaging of all data points. This is the result of heavier weighting of refinery corrosion measurements relative to short time laboratory results. Also evident from a comparison of the correlation and summary graphs is that the bands in the former are narrower than the spread of original data points in Figures 1, 2 and 3. Although the upper and lower limits of the corrosion bands are set on a fairly arbitrary basis, there is justification for telescoping the data into a narrower range, since the data are not individually corrected for variations in environmental conditions and, in addition, are subject to inherent error. In practical applications of the correlation graph (Figure 4), the width of these bands may be loosely interpreted as a temperature parameter.

It is estimated that the corrosion rates predicted by the correlation graph (Figure 4) are correct within a factor of 2, with the highest level of confidence in the medium hydrogen sulfide levels for carbon and chrome-moly steel. This degree of accuracy is sufficient for most engineering purposes and the correlation is therefore considered to offer a useful design tool for choosing suitable materials of construction for catalytic reforming equipment.

General Review of Catalytic Reformer Corrosion and Materials

Characteristics of Hydrogen Sulfide Corrosion

Elevated temperature corrosion by hydrogen sulfide experienced in catalytic reforming (and also catalytic desulfurizing) units differs in two important aspects from the familiar sulfide attack commonly encountered in other types of refinery processes where the sulfur is predominantly in the form of organic sulfur compounds. First, there is the markedly greater corrosiveness of hydrogen sulfide relative to such sulfur compounds as mono- and disulfides, mercaptans and thiophenes. Note the high corrosion rate (approximately 100 mils per year) predicted by Figure 4 for 0.1 volume percent hydrogen sulfide at 850-1050 F; in contrast, an equivalent amount of organically combined sulfur ordinarily has negligible corrosive action.

The second, and perhaps the most distinguishing, characteristic of hydrogen sulfide corrosion is the proved lack of superior corrosion resistance of the chrome-moly steels as compared with carbon steel. This fact is remarkable considering the outstanding performance of these steels in withstanding all other forms of sulfide attack on conventional types of refinery units processing sour stocks.

Characteristics of Sulfide Scales

The sulfide scale formations commonly found in the high temperature portions of catalytic reforming units are bulky, brittle and porous, and have a general tendency to spall off. As previously mentioned, these physical properties render them generally ineffectual as barriers to stifling further corrosion, although isolated instances are on record where the scale formed a dense and adherent layer which protected the underlying metal.

Experience indicates that the operational difficulties caused by scaling frequently outweigh the detriment of metal loss. Once dislodged, the scale has the tendency to crumble into a fine powder which plugs up processing equipment, predominantly the catalyst beds. The resultant high pressure drops frequently necessitate unscheduled shut-downs for catalyst screening. Another operational difficulty associated with scaling is its insulating effect in furnace tubes which may lead to local overheating and subsequent rupture.

Other Forms of Catalytic Reformer Corrosion

A discussion of corrosion problems experienced in catalytic reforming units would be incomplete without brief mention of high temperature hydrogen attack. This topic is of special interest in view of the recent cracking failure of three catalytic reformer reactors which was ascribed to this type of damage. As described by Ciuffreda and Rowland,[9] the failure occurred as the result of insulation breakdown which permitted the hot hydrogen rich gases to come in contact with the carbon steel shell causing decarburization and embrittlement. Fortunately, this form of metal deterioration is fairly well understood and can be readily prevented by the choice of proper materials as set forth by Nelson.[10] For the temperatures and hydrogen partial pressures ordinarily encountered in catalytic reforming processes, 1 percent Cr-½ percent Mo steels furnish adequate resistance to hydrogen attack. C-½ percent Mo and ½ percent Cr-½ percent Mo steel also are suitable for portions of the unit operating at temperatures lower than the reactors and furnaces.

Low temperature galvanic or electrochemical corrosion ordinarily is not a problem in the majority of catalytic reforming processes. This is because only trace quantities of liquid water are present under normal operating conditions. On those units where water is present, instances of fairly serious corrosion occasionally are experienced in and around the reactor effluent condenser and separator drum. This corrosion is attributed to dilute hydrochloric acid which is formed from chlorides in the feed or injected for catalyst rejuvenation. Acidic condensate corrosion also may be a problem during the regeneration cycle (in units practicing "in situ" catalyst regeneration) unless precautions are taken to maintain the oxidizing gas mixture above its dew point.

[6] The scale factor measures the bulkiness of the scale and is numerically equal to the scale thickness divided by the corresponding metal loss. In the monosulfide scales commonly formed on low alloy steels in hot H_2:H_2S atmospheres, the observed scale factor ranges from 2½ to 4.

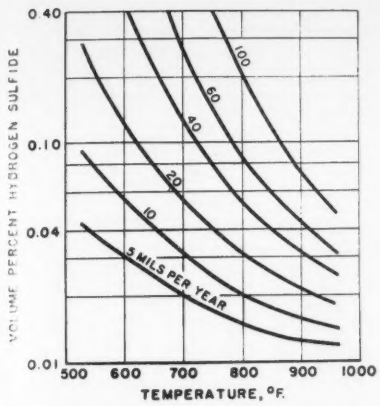

Figure 5—Lines of constant corrosion rate for carbon and low chrome (up to 5 percent Cr) steels versus temperature and volume percent hydrogen sulfide in Catforming reaction gas mixture.

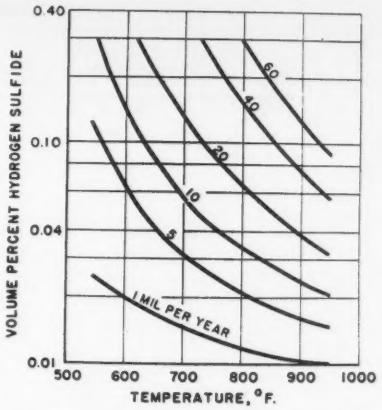

Figure 6—Lines of constant corrosion rate for 11½ to 13½ chrome steel versus temperature and volume percent hydrogen sulfide in Catforming reaction gas mixture.

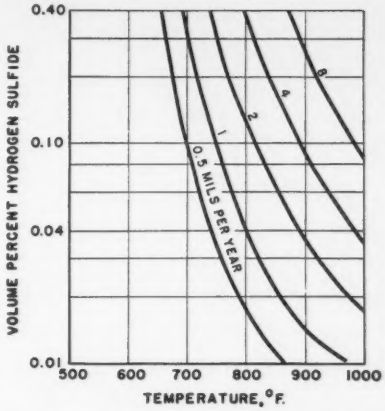

Figure 7—Lines of constant corrosion rate for 18 chrome-8 nickel austenitic stainless steel versus temperature and volume percent hydrogen sulfide in Catforming reaction gas mixture.

Under these circumstances, the corrosion is attributed to dilute sulfurous or sulfuric acid formed by the hydrolysis of sulfur oxides in the regeneration gases.

Another form of low temperature attack occasionally found in catalytic reforming equipment is characterized by cracking failures of brasses and austenitic stainless steels. There is evidence indicating that this attack is experienced only during downtime conditions. Cracking of brass heat exchanger tubes has occurred in a number of instances during lengthy shutdown periods. These failures are clear-cut examples of stress corrosion cracking and are attributed to the combined action of ammonia, oxygen and moisture. Cracking failures of austenitic stainless steels in catalytic reformer furnace tubes are reported in the literature (Reference 11). It is speculated that the corrosive agents responsible for this attack are polythionic acids formed by oxidation and hydrolysis of iron sulfide scale. At this time it is not established whether these failures are the result of stress corrosion cracking, intergranular attack or a combination of these two corrosion phenomena.

Solutions to the Corrosion Problem

As in almost any corrosion problem, the available counter-measures involve either changing the corrosive environment or employing more resistant materials of construction. The former approach entails reducing the amount of hydrogen sulfide since the temperature is essentially fixed by process considerations. This can be achieved by scrubbing the recycle gas in a recirculating amine absorption system or by desulfurizing the feed stock in a cobalt-moly catalytic pretreater.

Amine treating is only partially effective in reducing the level of corrosion in the unit. This is because while preventing build-up of hydrogen sulfide in the recycle gas, it does not protect the reactor train from corrosion by the hydrogen sulfide produced during the once through passage of the feed naphtha. Catalytic feed desulfurization obviously is more satisfactory from a corrosion prevention point of view since it effects nearly complete removal of hydrogen sulfide producing compounds from the fresh feed and thus minimizes corrosion in all parts of the unit. Consideration generally should be given to amine treating or predesulfurizing for units processing naphthas containing in excess of 0.01 weight percent sulfur; for sulfur levels above 0.05 weight percent, pretreating appears to be the more practical method. In the final analysis, the desirability for or choice between either of these installations depends on an economic study in which investment and operating costs are balanced against savings in reformer materials and improvements of product quality.

Ferrous Materials of Construction

The most widely employed materials of construction for catalytic reforming plants are the low chrome-moly steels required for resistance to hydrogen attack. The earlier units have a proportionately larger amount of 5 percent Cr-½ percent Mo equipment because this material was initially believed to furnish better protection against hydrogen sulfide corrosion. In more recent construction and in new designs, the 1 percent, 1¼ percent and 2¼ percent chrome-moly steels are more heavily favored, the specific choice being influenced by price availability, mechanical properties (high temperature strength), as well as by the general preference of the individual refiners. C-½ percent Mo and ¼ percent Cr-½ percent Mo steels are not widely used in catalytic reformer construction; in addition to their more limited resistance to hydrogen attack and graphitization, they are not as readily available.

Where the level of hydrogen sulfide is sufficiently high to cause excessive corrosion of these steels, more resistant materials are employed. The dividing line predicated on corrosion rate falls generally into the 15-50 mils per year range, depending on the part in question and naturally also on the engineering philosophy of the company operating the unit. Among the higher alloy materials, the various grades of austenitic Cr-Ni steels, principally AISI Type 304 (18 percent Cr/8 percent Ni), find wide application. The straight 11-13 percent Cr steels also are employed but there is a growing tendency to go directly to the 18 percent Cr/8 percent Ni compositions which, while higher priced than the 11-13 percent Cr steels, offer a considerably greater and more reliable degree of corrosion resistance.

Aluminum Coatings

In an effort to find more economical substitutes for stainless steel, many refiners have turned to a variety of aluminum surface coatings for protecting catalytic reforming equipment for high temperature hydrogen sulfide attack. Three different types of aluminum coatings are being used commercially — dipped, sprayed (with or without post diffusion treatment) and diffused (calorized). These coatings generally are applied to low alloy steels because the aluminum layer is not considered to protect the underlying metal from high temperature hydrogen attack. Experience indicates that all forms of aluminum coatings can furnish excellent corrosion resistance provided the aluminum remains as a continuous coating firmly bonded to the steel.

The longest service experience on record is reported by the Standard Oil Company of Indiana.[2] In one of their catalytic reformer units, dipped aluminum coatings in service since 1954 still show no evidence of significant visible deterioration. More recent installations of internally sprayed and diffused furnace tubes are giving satisfactory performance after more than one year's operation. These and other encouraging reports on the effectiveness of soundly applied aluminum coatings foreshadow their more extensive use for future catalytic reformer construction.

Acknowledgment

This survey report was prepared under the sponsorship of the National Association of Corrosion Engineers (Committees T-8 and T-5B-2). The author wishes to express his appreciation to the various oil companies for both furnishing and permitting the release of hitherto unpublished data.

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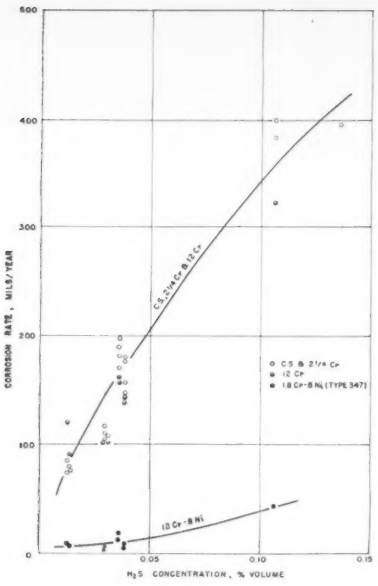

Figure 8—Effect of hydrogen sulfide concentration on corrosion of steel in commercial Hydroforming unit.

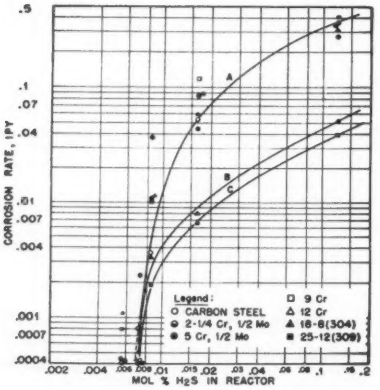

Figure 9—Corrosion rate versus hydrogen sulfide concentration under catalytic reforming conditions at 1000 F.

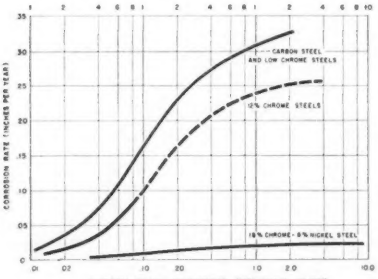
CORROSION RATE (INCHES PER YEAR)
HYDROGEN SULFIDE CONCENTRATION (PER CENT BY VOLUME)
CARBON STEEL AND LOW CHROME STEELS
12% CHROME STEELS
18% CHROME - 8% NICKEL STEEL

Figure 10—Anticipated maximum corrosion rates in catalytic-reforming equipment. Temperature was 950 F; pressure was over 400 psig.

TABLE 2

Location in Unit	Temperature (Deg. F)	Pressure (Psig)	Material	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	18 Cr/8 Ni
Recycle Gas Furnace (Outlet)	1025	400	C.S.	50	—	—
			1¼ Cr	64	—	—
			2¼ Cr	72	—	—
			5 Cr	76	—	—
			9 Cr	96	—	—
			12 Cr	—	28	—
			Type 304	—	—	20
D-2 Reactor (Inlet)	950	400	C.S.	46	—	—
			1¼ Cr	40	—	—
			2¼ Cr	30	—	—
			5 Cr	92	—	—
			9 Cr	36	—	—
			12 Cr	—	0	—
			Type 304	—	—	10
D-6 Reactor (Outlet)	950	265	C.S.	62	—	—
			1¼ Cr	60	—	—
			2¼ Cr	62	—	—
			5 Cr	58	—	—
			9 Cr	40	—	—
			12 Cr	—	62	—
			Type 304	—	—	8
Average Corrosion Rates:				59	30	13

H_2S Conc.: 0.03-0.07 vol. percent; Duration: 89 days.

TABLE 3

Location in Unit (Furnace)	Duration (Days)	Pressure (Psig)	Material	Corrosion Rate (Mils/Year)
B-2 Recycle Gas	89	400	1¼ Cr	39
			2¼ Cr	65
B-4 Reheat	54	365	1¼ Cr	15
	89			58
B-5 Reheat	54	330		31
	89			77
B-6 Reheat	36	300		39
	89			70
B-7 Reheat	54	265		43
	89			149
Average Corrosion Rate:				59

Temperature: 950 F; H_2S Conc.: 0.03-0.07 vol. percent.

TABLE 4

Corrosion Rate (Mils/Year)	H_2S Concentration (Vol. Percent) 0-5 Cr	11½-13½ Cr	18 Cr/8 Ni
1	—	0.0105	0.015
2	—	—	0.036
4	—	—	0.090
5	0.013	0.017	—
8	—	—	0.27
10	0.0165	0.024	—
20	0.0215	0.039	—
40	0.031	0.075	—
60	0.043	0.13	—
100	0.074	—	—

Temperature: 900 F; Pressure: 500 psi; Duration: 1-356 days.

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13. Canadian Petrofina Ltd. (Aug. 30, 1956).*
14. Major U. S. Refining Company (May 3, 1957).(7)*
15. Major West Coast Refining Company (Dec. 10, 1956).(7)*
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21. Texas Company (Feb. 4, 1957).*
22. A. Dravnieks and C. H. Samans (Standard Oil Company of Indiana). Kinetics of the Reaction of Steel with Hydrogen Sulfide-Hydrogen Mixtures. Presented at the 110th Meeting of the Electrochemical Society, Cleveland, Ohio (October 3, 1956).

(7) Company name withheld on request.
(8) Now International Nickel Company.
* Private communication.

TABLE 5

Location	Material	H_2S Conc. (Vol. %)	Duration (Days)	Corrosion Rate (Mils/Year)
Port Arthur, Texas	2¼ Cr-1 Mo	0.036	180	38
Philadelphia, Pa.	12 Cr	0.016	180	7

Temperature: 900 F; Pressure: 500 psig.

TABLE 6

Material	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	18 Cr/8 Ni
C.S.	21	—	—
1 Cr	18	—	—
2¼ Cr	13	—	—
5 Cr	19	—	—
9 Cr	6.8	—	—
12 Cr	—	8.0	—
Type 304	—	—	2.4
Type 316	—	—	2.5
Av. Corr. Rates	16	8	2.5

Temperature: 910 F; Pressure: 310 psig; H_2S Conc.: 0.008 vol. percent; Duration: 90 days.

TABLE 7

Location in Unit	Temperature (Deg. F)	H_2S Conc. (Vol. %)	Duration (Hours)	Corrosion Rate (Mils/Year) C.S.	2¼ Cr	12 Cr	18 Cr/8 Ni
Recycle Gas Furnace Outlet	947	0.0125	23	72.9	84.9	119.7	9.4
	928	0.0387	96	175.0	180.4	140.2	5.6
	956	0.036	72	180.6	197.1	156.9	18.8
	908	0.0294	24	105.0	110.0	101.9	6.9
	937	0.1059	48	384.1	400.4	323.3	42.1
Last Reheat Furnace Outlet	940	0.0136	72	74.5	78.8	90.7	6.7
	901	0.0387	96	144.4	155.2	136.8	9.4
	912	0.036	72	168.7	188.5	154.3	12.8
	900	0.0294	24	116.5	108.0	102.9	5.1
	930	0.1357	96	398.0	—	—	—

Pressure: 300 psig.

TABLE 8

Equipment	Material	Temperature (Deg. F)	Corrosion Rate (Mils/Year)
Furnace Tubes	C.S. and 2¼ Cr	900–1025	200 (max.)
Piping	1¼ Cr	850– 975	90 (max.)

H_2S Conc.: 0.035 vol. percent*; Pressure: 300 psig; Duration: 127 days.

* Based on an average of 0.02 weight percent sulfur in the naphtha charge (corresponding H_2S concentration of 0.035 vol. percent is obtained from Figure 6 of the reference paper, Reference 6).

TABLE 9

Duration (Days)	Material	Corrosion Rate (Mils/Year) 2¼—5 Cr	12 Cr
226	2¼ Cr	15	—
417		14	—
577		14	—
226	5 Cr	13	—
417		13	—
577		12	—
226	12 Cr	—	12
417		—	11
577		—	10
Av. Corrosion Rates:		13.5	11.0

Temperature: 920-960 F; Pressure: 500-535 psig; H_2S Conc.: 0.035 vol. percent*.

* Calculated from sulfur in feed naphtha and H_2S content in recycle gas by method presented in Reference 3 (Appendix I). A molecular weight of 120 for the naphtha and a 10:1 molal recycle-feed ratio is assumed.

TABLE 10

Duration (Days)	Material	Corrosion Rate (Mils/Year) 2¼ Cr-5 Cr	12 Cr	18 Cr/8 Ni
139	2¼ Cr	43	—	—
232		43	—	—
348		43	—	—
139	5 Cr	43	—	—
232		38	—	—
348		39	—	—
139	12 Cr	—	31	—
232		—	26	—
348		—	22	—
139	Type 304	—	—	4
232		—	—	4
348		—	—	3
Av. Corrosion Rates:		41.5	26.3	3.7

Temperature: 900-920 F; Pressure: 500-535 psig; H_2S Conc.: 0.08 vol. percent*

* Calculated from sulfur in feed naphtha and H_2S content in recycle gas by method presented in Reference 3 (Appendix I). A molecular weight of 120 for the naphtha and a 10:1 molal recycle-feed ratio is assumed.

TABLE 11

Material	Corrosion Rate (Mils/Year) 2¼ Cr—5 Cr	12 Cr
2¼ Cr	39	—
5 Cr	34	—
12 Cr	—	27
Av. Corr. Rates:	36.5	27

Pressure: 500-535 psig; H_2S Conc.: 0.09 vol. percent*; Duration: 234 days; Temperature: 875-900 F.

* Calculated from sulfur in feed naphtha and H_2S content in recycle gas by method presented in Reference 3 (Appendix I). A molecular weight of 120 for the naphtha and a 10:1 molal recycle-feed ratio is assumed.

TABLE 12

Location in Unit	Temperature (Deg. F)	Pressure (Psig)	Material	Corrosion Rate (Mils/Year) 0-5 Cr	12 Cr	18 Cr/8 Ni
No. 1 Reactor (Inlet)	915–920	565	5 Cr	20.6	—	—
			12 Cr	—	7.9	—
			Type 302	—	—	1.5
			Type 316	—	—	6.3
			Type 347	—	—	1.2
No. 4 Reactor (Inlet)	900–910	540	C.S.	15.6	—	—
			2¼ Cr	18.8	—	—
			5 Cr	20.2	—	—
			12 Cr	—	8.7	—
			Type 302	—	—	6.8
			Type 316	—	—	6.3
			Type 347	—	—	4.6
Average Corrosion Rates:				18.9	8.3	4.5

H_2S Conc.: 0.015 vol. percent; Duration: 72 days.

DATA SECTION

American Oil Company

"Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data" (Reference 12).

Commercial Unit Corrosion Test

Corrosion rates obtained in Ultraformer at Texas City, Texas are presented in Table 2. These data appear in Table 22 of the reference paper (Reference 12).

Inspection of Commercial Equipment

Corrosion rate measurements obtained from tubes in the radiant sections of the preheat, reheat and recycle gas furnaces of the Ultraformer at Texas City, Texas are summarized in Table 3. These data are a revised version of those originally appearing in Table 21 of the reference paper (Reference 12).

Atlantic Refining Company

1. "Selection of Materials for Construction of Catalytic Reforming Units" (Reference 7).

Correlation Curves

The iso-corrosion curves reproduced in Figures 5, 6 and 7 were prepared from correlations of laboratory and pilot plant test data supplemented by data from plant tests and measurements of metal loss of operating plant equipment (up to one year's operation). The laboratory results were obtained in tests of 24 hours duration with H_2S:H_2 mixtures at 900 F and 500 psig.

Curves ATL in summary graphs (Figures 1, 2 and 3) are derived by cross-plotting the corrosion rates from the iso-corrosion curves (Figures 5, 6 and 7) at 900 F. The individual values of corrosion rate thus obtained are listed in Table 4.

2. "Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data" (Reference 12).

Inspection of Commercial Equipment

Corrosion rates obtained from Catformer units in Port Arthur, Texas and Philadelphia, Pa., are tabulated in Table 5. These data are taken from Table 23 of the reference paper (Reference 12).

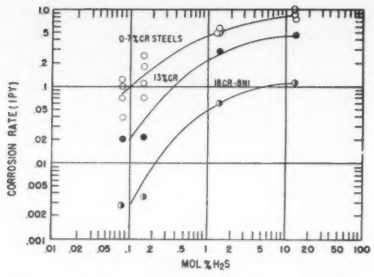

Figure 11—Corrosion rates of various steels in hydrogen sulfide-hydrogen mixtures at 950 F, 500 psi, 100-hour laboratory tests.

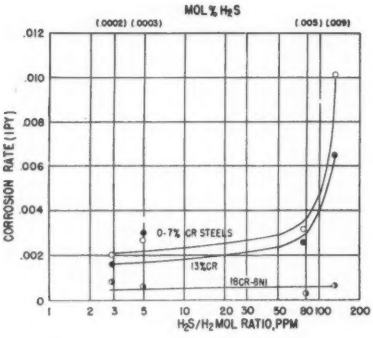

Figure 12—Catalytic reformer average corrosion rates at 925 F, average 500 psi.

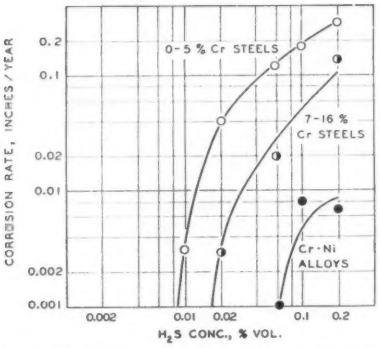

Figure 13—Effect of hydrogen sulfide concentration on corrosion rate at 985 F and 485 psig hydrogen pressure.

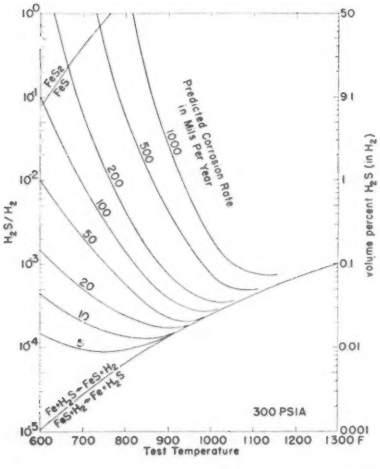

Figure 14—Diagram for predicting corrosion rates of low-carbon steels by mixtures of hydrogen sulfide and hydrogen at a total pressure of 300 psia.

TABLE 13

Material	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	18 Cr/8 Ni
C.S.	32	—	—
1¼ Cr	—	—	—
2¼ Cr	—	—	—
5 Cr	38	—	—
7 Cr	37	—	—
9 Cr	34	—	—
12 Cr	—	15	—
18 Cr/8 Ni	—	—	4.3
Av. Corr. Rates	35	15	4.3

Temperature: 925 F; Pressure: 680 psig; H_2S Conc.: 0.05 vol. percent; Duration: 148 days.

TABLE 14

Material	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	18 Cr/8 Ni
C.S.	47	—	—
1¼ Cr	47	—	—
2¼ Cr	50	—	—
5 Cr	47	—	—
7 Cr	44	—	—
9 Cr	—	—	—
12 Cr	—	22	—
18 Cr/8 Ni	—	—	7.2
Av. Corr. Rates	47	22	7.2

Temperature: 925 F; Pressure: 680 psig; H_2S Conc.: 0.063 vol percent*; Duration: 251 days.

* The corrosion rates listed in Table 14 are cumulative values corresponding to 251 days exposure at a weighted average H_2S concentration of 0.063 vol. percent. The *actual* H_2S conc. during run No. 2 (103 days) was 0.08 vol. percent.

TABLE 15

Location in Unit	Material	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr
Reactor 601	C.S.	17	—
	1 Cr	18	—
	2 Cr	18	—
	5 Cr	20	—
	7 Cr	17	—
	9 Cr	19	—
	12 Cr	—	8
Reactor 602	C.S.	17	—
	1 Cr	18	—
	2 Cr	18	—
	5 Cr	20	—
	7 Cr	17	—
	9 Cr	19	—
	12 Cr	—	8
Reactor 603	C S	16	—
	1 Cr	17	—
	2 Cr	18	—
	5 Cr	20	—
	7 Cr	18	—
	9 Cr	20	—
	12 Cr	—	8
Reactor 604	C.S.	14	—
	1 Cr	17	—
	2 Cr	18	—
	5 Cr	19	—
	7 Cr	16	—
	9 Cr	19	—
	12 Cr	—	8
Average Corrosion Rates:		18	8

Temperature: 950 F; Pressure: 625 psig; H_2S Conc.: 0.015 vol. percent; Duration: 71 days.

Canadian Petrofina Ltd.

Private Communication (Reference 13).

Commercial Unit Corrosion Test

Corrosion rates obtained from test coupons (supplied by the International Nickel Company) exposed in the first reactor of the Ultraformer at Montreal, P.Q., Canada are summarized in Table 6.

D-X Sunray Oil Company

"Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data" (Reference 12).

Inspection of Commercial Equipment

A corrosion rate of 40 mils per year was measured on a 5 Cr reactor effluent exchanger tube after six months service in the Catformer at Tulsa, Okla. The temperature, pressure and H_2S content were respectively 870 F, 400 psig and 0.04 vol. percent. These data appear in Table 29 of the reference paper (Reference 12).

Humble Oil and Refining Company

"High Temperature Sulfide Corrosion in Catalytic Reforming of Light Naphthas" (Reference 6).

(a) *Commercial Unit Corrosion Test*

Corrosion rates measured in the Hydroformer unit at Baytown, Texas are presented in Table 1 of the reference paper (Reference 6). An extract of pertinent data are summarized in Table 7 and Figure 8, and appear as Curves HUM-1 in the summary graphs (Figures 1, 2 and 3).

(b) *Inspection of Commercial Equipment*

Inspection of preheat and reheat furnace tubes and of hot piping on the No. 2 Catalytic Reforming Unit at Baytown, Texas, yielded the results summarized in Table 8.

(c) *Pilot Plant Corrosion Tests*

Corrosion rates obtained in 96 hour pilot plant tests at 1000 F, reproduced in Figure 9, appear in the summary graphs (Figures 1, 2 and 3) as Curves HUM-2.

Major U. S. Refining Company

Confidential Communication (Reference 14).

Commercial Unit Corrosion Tests

Corrosion rates obtained from test spools exposed in Platformer reactors are summarized in Tables 9, 10 and 11.

Major West Coast Refining Company

Private Communication (Reference 15).

Commercial Unit Corrosion Tests

Corrosion rates obtained from test spools exposed in catalytic reforming reactors are summarized in Table 12.

TABLE 16

Run No.	Duration (Days)	Cumulative Exposure (Days)	H_2S Conc. (Vol. %)	Overall Corrosion Rate (Mils/Year)
1	148	148	0.05	135*
2	103	251	0.08	114*
3	69	320		98
4	64	384		82
5	97	481		65
6	81	562		71
7	62	624		69
8	134	758		56*

Temperature: 950 F; Pressure: 680 psig.

* Only the three rates corresponding to exposures ending with runs Nos. 1, 2 and 8 are plotted in the summary graph (Figure 1).

TABLE 17

Equipment	Temperature (Deg. F)	Duration (Days)	Corrosion Rate (Mils/Year) 5 Cr	12 Cr
Piping	900–950	120 270	7 10	— —
Furnace Tube	975–1025	270	10	—
Reactor Internal	900–950	120 270	— —	5 8
Average Corrosion Rates			9.0	6.5

Pressure: 500 psig; H_2S Conc.: 0.005 vol. percent.

TABLE 18

Location In Unit	Temperature (Deg. F)	Corrosion Rate (Mils/Year) 0-9 Cr	12-16 Cr	Cr-Ni
Reactor (Top)	885	83	32	10
Reactor (Bottom)	935	120	13	3
Reactor Effluent	905	120	20	3
Average Corrosion Rates:		108	21	5

Pressure: 175 psig; H_2S Conc.: 0.5 vol. percent Duration: 55 days.

TABLE 19

Location In Unit	Temperature (Deg. F)	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	Cr-Ni
No. 1 Reactor (Inlet)	910	64	26	4
No. 2 Reactor (Inlet)	895	62	24	5
No. 3 Reactor (Inlet)	885	60	20	4
Average Corrosion Rates:		62	23	4.3

H_2S Conc.: 0.04 vol. percent; Pressure: 550 psig; Duration: 50.5 days.

TABLE 20

Location In Unit	Temperature (Deg. F)	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	Cr-Ni
No. 1 Reactor (Inlet)	910	30	15	2
No. 2 Reactor (Inlet)	920	28	18	1.5
No. 3 Reactor (Inlet)	915	28	16	1
Average Corrosion Rates:		29	16	1.5

H_2S Conc.: 0.02 vol. percent; Pressure: 570 psig; Duration: 213 days.

TABLE 21

Location In Unit	Temperature (Deg. F)	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	Cr-Ni
No. 1 Reactor (Inlet)	900	19	14	1
No. 2 Reactor (Inlet)	905	18	13	0.5
No. 3 Reactor (Inlet)	900	18	12	0.5
Average Corrosion Rates:		18	13	0.7

H_2S Conc.: 0.018 vol. percent; Pressure: 575 psig; Duration: 212 days.

TABLE 22

H_2S Concentration (Vol. Percent)	Corrosion Rate (Mils/Year) 0-9 Cr	12 Cr	18 Cr/8 Ni
0.013	10	4	1.3
0.017	20	8	2.5
0.024	50	20	6.3
0.040	100	40	12.5
0.075	200	80	25.0
0.44	500	200	62.5

Temperature: 900 F; Pressure: 285 psig; Duration: 0.7-11.7 days.

TABLE 23

Location in Unit (Plant A)	Material	Corrosion Rate (Mils/Year) 0-5 Cr	12 Cr	18 Cr/8 Ni
Reactor 2B	C.S.	0.13	—	—
	1¼ Cr	0.87	—	—
	2¼ Cr	1.36	—	—
	5 Cr	0.93	—	—
	12 Cr	—	0.83	—
	Type 304	—	—	0.09
Average Corrosion Rates		0.82	0.83	0.09

Temperature: 925-960 F; Pressure: 300-500 psig; H_2S Conc.: 0.012 vol. percent; Duration: 258 days.

Pure Oil Company

"Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data" (Reference 12).

Inspection of Commercial Equipment

A corrosion rate of 33 mils per year was estimated for a 2 Cr preheat furnace tube after eight months of operation in the Sinclair-Baker catalytic reformer at Heath, Ohio. The approximate conditions were 900-1000 F skin temperature, 500 psig pressure and 0.026 vol. percent H_2S.

Richfield Oil Corporation

"How Richfield Plans to Combat High-Temperature Sulfide Corrosion in Its New Catalytic Reformer" (Reference 16).

Correlation Curves

The corrosion rate curves reproduced in Figure 10 and as Curves RIC in the summary graphs (Figures 1, 2 and 3) predict maximum corrosion rates at 950 F and 400 psig. These curves represent a composite of 113 data points from six different oil companies covering a range of laboratory, pilot plant and refinery corrosion tests, as well as measurements on operating equipment. The corrosion rates are pro-rated to 400 hours exposure in accordance with the findings by Backensto et al (Reference 5).

Shell Oil Company

1. "Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data" (Reference 12).

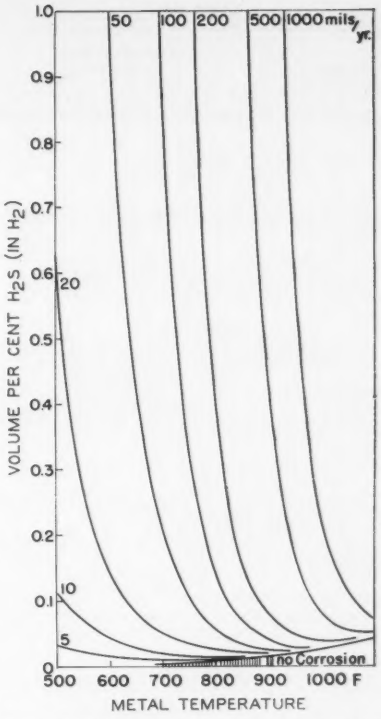

Figure 15—Diagram similar to Figure 14, but plotted on a linear percent by volume hydrogen sulfide (in pure hydrogen) scale—total pressure of 300 psia.

Commercial Unit Corrosion Tests

Corrosion rates obtained in the Platformer unit at Wood River, Ill., are summarized in Tables 13 and 14, respectively for run No. 1 and runs Nos. 1 and 2 combined. These data are a revised version of those originally appearing in Table 31 of the reference paper (Reference 12).

2. Private Communication (Reference 17).

(a) *Commercial Unit Corrosion Tests*

Corrosion rates obtained in the Platformer unit at Anacortes, Wash., are summarized in Table 15.

(b) *Inspection of Commercial Equipment*

Periodic inspection of a spool piece from a 5 Cr preheat furnace transfer line in the No. 1 Platformer unit at Wood River, Ill., yielded the results listed in Table 16.

A corrosion rate of 68 mils/year was measured on a sectioned 9 Cr reheat furnace tube from the No. 1 Platformer unit at Wood River, Ill. The tube had been exposed 758 days to 0.08 vol. percent H_2S at 670 psig and 955 F (1135 F max. metal temperature).

Sinclair Refining Company

1. "Effect of Hot H_2S Environments on Various Metals" (Reference 4).

(a) *Laboratory Corrosion Tests*

Corrosion rates obtained in 100 hour laboratory tests with H_2S:H_2 mixtures at 950 F and 500 psig are reproduced in Figure 11, and appear in the summary graphs (Figures 1, 2 and 3) as Curves SIN-1.

TABLE 24

Location in Unit (Plant A)	Material	Corrosion Rate (Mils/Year) 0-5 Cr	12 Cr	18 Cr/8 Ni
Reactor 1A	C.S.	5.1	—	—
	1¼ Cr	6.6	—	—
	2¼ Cr	6.7	—	—
	5 Cr	6.1	—	—
	12 Cr	—	5.8	—
	Type 304	—	—	0.7
Average Corrosion Rates		6.1	5.8	0.7

Temperature: 925-960 F; Pressure: 300-500 psig; H_2S Conc.: 0.017 vol. percent; Duration: 395 days.

TABLE 25

Location in Unit (Plant B)	Material	Corrosion Rate (Mils/Year) 0-5 Cr	12 Cr	18 Cr/8 Ni
Reactor 101	C.S.	21.6	—	—
	1¼ Cr	30.7	—	—
	2¼ Cr	33.3	—	—
	5 Cr	27.7	—	—
	12 Cr	—	9.0	—
	Type 304	—	—	2.1
	Type 316	—	—	1.1
Average Corrosion Rates		28.3	9.0	1.6

Temperature: 925-960 F; Pressure: 525-625 psig; H_2S Conc.: 0.031 vol. percent; Duration: 188 days.

TABLE 26

Location in Unit (Plant B)	Material	Corrosion Rate (Mils/Year) 0-5 Cr	12 Cr	18 Cr/8 Ni
Reactor 101	C.S.	24.3	—	—
	1¼ Cr	32.1	—	—
	2¼ Cr	35.0	—	—
	5 Cr	31.2	—	—
	12 Cr	—	12.1	—
	Type 304	—	—	2.7
	Type 316	—	—	2.7
Reactor 103	C.S.	29.4	—	—
	1¼ Cr	31.5	—	—
	2¼ Cr	26.5	—	—
	5 Cr	27.9	—	—
	12 Cr	—	11.2	—
	Type 304	—	—	1.2
	Type 316	—	—	1.1
Average Corrosion Rates		29.7	11.7	1.9

Temperature: 925-960 F; Pressure: 525-625 psig; H_2S Conc.: 0.040 vol. percent; Duration: 289 days.

(b) *Commercial Unit Corrosion Tests*

Corrosion rates obtained from test spools placed at various locations in the catalytic reforming units at Marcus Hook, Pa., and East Chicago, Ind., are summarized in Table 4 of the reference paper (Reference 4). A graphical presentation of these data is reproduced in Figure 12 and also appears as Curves SIN-2 in the summary graphs (Figures 1, 2 and 3).

2. "Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data" (Reference 12).

Inspection of Commercial Equipment

Corrosion rates obtained from operating catalytic reforming equipment at Marcus Hook, Pa., are summarized in Table 17. These data are taken from Table 32 of the reference paper (Reference 12).

Socony Mobil Oil Company

1. "High Temperature Hydrogen Sulfide Corrosion" (Reference 5).

TABLE 27

Location in Unit	Duration (Days)	Material	Corrosion Rate (Mils/Year) 0-5 Cr	12 Cr	18 Cr/8 Ni
Reactor 1 (Inlet)	208	C.S.	2.1	—	—
		C–½ Mo	3.4	—	—
		1 Cr	3.7	—	—
		2 Cr	3.7	—	—
		2¼ Cr	4.2	—	—
		5 Cr	3.9	—	—
		12 Cr	—	2.3	—
		18 Cr/8 Ni	—	—	0.3
Reactor 4 (Inlet)	208	2¼ Cr	5.3	—	—
		5 Cr	3.8	—	—
		18 Cr/8 Ni	—	—	0.2
	150	C.S.	4.6	—	—
		5 Cr	2.9	—	—
	282	C.S.	2.3	—	—
		2 Cr	1.7	—	—
		5 Cr	1.0	—	—
		18 Cr/8 Ni	—	—	0.8
	133	C.S.	2.3	—	—
Reactor Prods. Exchanger	668	C.S.	2.2	—	—
		C–½ Mo	2.2	—	—
		1 Cr	2.4	—	—
		2 Cr	2.1	—	—
		2¼ Cr	2.7	—	—
		5 Cr	2.1	—	—
		12 Cr	—	1.5	—
		18 Cr/8 Ni	—	—	0.1
Average Corrosion Rates:			2.9	1.9	0.3

Temperature: 940 F; Pressure: 600 psig; H_2S Conc.: 0.0016 vol. percent.

Laboratory Corrosion Tests

Corrosion rates obtained in laboratory tests lasting up to 500 hours in H_2S:H_2 mixtures at 985 and 485 psig are reproduced in Figure 13, and appear in the summary graphs (Figures 1, 2 and 3) as Curves SOC.

A corrosion rate of 44 mils/year was measured on a 5 Cr laboratory furnace tube after a three week run with Kuwait naphtha mixed with 11.1 vol. percent H_2 and 0.085 vol. percent H_2S. The pressure and tube skin temperature were 460 psig and 905 F respectively.

2. "High Temperature Hydrogen Sulfide Corrosion in Thermofor Catalytic Reformers" (Reference 18).

Commercial Unit Corrosion Tests

Corrosion rates measured on test coupons exposed for 55 days in the Thermofor Catalytic Reforming unit at Torrance, California, are tabulated in Table 18. These data are presented in Figure 3 of the reference paper (Reference 18).

3. "High-Temperature Hydrogen Sulfide Corrosion in Commercial Sovaformer Units" (Reference 19).

Commercial Unit Corrosion Tests

Corrosion rates measured on test coupons exposed in the Sovaformer unit at Ferndale, Wash., are tabulated in Tables 19 and 20. These data are taken from Figures 2 and 4 of the reference paper (Reference 19).

Corrosion rates measured on test coupons exposed in the Sovaformer unit at Augusta, Kan. are tabulated in Table 21. These data are taken from Figure 9 of the reference paper (Reference 19).

Standard Oil Company of Indiana

1. "Corrosion Control in Ultraforming" (Reference 2).

(a) *Laboratory Corrosion Tests*

Figures 14 and 15 represent isocorrosion curves for low carbon steels obtained in laboratory tests up to 280 hours duration with H_2S:H_2 mixtures. According to the experimenters, these results are equally applicable to steels containing up to approximately 9 percent Cr.

Curve IND in summary graph (Figure 1) is derived by cross-plotting the corrosion rates from Figure 15 at 900 F. Curves IND for 12 Cr and 18 Cr/8 Ni steels, appearing in the other summary graphs (Figures 2 and 3), are constructed by dividing the corrosion rate values of Figure 15 by 2.5 and 8.0 respectively, in accordance with Indiana's experimental findings.

The individual values of corrosion rate thus obtained are listed in Table 22.

(b) *Commercial Unit Corrosion Tests*

Corrosion rates of 45 and 70 mils per year were obtained from carbon steel coupons exposed in the recycle gas stream of an Ultraformer unit for 400-1100 hours. The respective approximate conditions of exposure were 950-1000 F, 0.32 vol. percent H_2S and 950 F, 0.23 vol. percent H_2S.

2. "Minutes of Meeting of N.A.C.E. Task Group T-5B-2" (Reference 3).

Commercial Unit Corrosion Test

Corrosion rates ranging from 32-43 mils per year on 0-9 Cr steels and 3.3 mils per year on 18 Cr/8 Ni steel were obtained in an 1125 hour run in the recycle gas furnace of the Ultraformer at Wood River, Ill. The coupons were exposed at a temperature and pressure of 1030 F and 340 psig in a gas containing 0.029 vol. percent H_2S.

Sun Oil Company

Private Communication (Reference 20).

Commercial Unit Corrosion Tests

Corrosion rates obtained from test spools exposed in the reactors of two Houdriformer units at Marcus Hook, Pa. are summarized in Tables 23, 24, 25 and 26.

Texas Company

Private Communication (Reference 21).

Commercial Unit Corrosion Tests

Corrosion rates obtained from test spools exposed in the No. 1 Platformer unit at Port Arthur, Texas, are summarized in Table 27.

Publication 58-3

NACE TECHNICAL COMMITTEE REPORT

High-Temperature Hydrogen Sulfide Corrosion of Stainless Steels★

A Contribution to the Work of NACE Technical Group Committee T-8 on Refinery Industry Corrosion, By E. B. Backensto,(1) R. E. Drew,(1) J. E. Prior(1) and J. W. Sjoberg(1)

Abstract

In recent years the petroleum industry has been plagued with a number of high temperature hydrogen sulfide corrosion problems, primarily in connection with catalytic reforming. To combat this problem from a standpoint of materials selection, only the austenitic steels offer satisfactory resistance to this type of attack under a wide range of conditions.

This paper reports the results of laboratory investigations chiefly on stainless steels, covering the following main topics: 1. The possible application of the new manganese-modified stainless steels, 2. The effect of heat treatment on corrosion rate, 3. A comparison of corrosion rates of wrought, weld and cast stainless steel, and 4. Some tests on stress corrosion cracking. 8.4.3

Introduction

HIGH-TEMPERATURE hydrogen sulfide corrosion problems in the petroleum industry became acute during the rapid expansion of catalytic reforming used to upgrade the octane number of motor gasolines. In catalytic reforming a large amount of hydrogen is present and the sulfur in the naphtha charge is converted to hydrogen sulfide. At the high temperatures in catalytic reformers the hydrogen sulfide attacks steels to form a troublesome iron sulfide scale which can interfere with continuous operation of the unit.

During the past few years the petroleum industry has been searching for practical solutions to high-temperature hydrogen sulfide corrosion problems. During this time Socony Mobil has been conducting an extensive study of this problem, including laboratory and field tests. Early data from this work were reported two years ago at the South Central Regional NACE Meeting.[1] Since that time a considerable amount of information on this subject has appeared also in the literature; a bibliography on this subject appeared in a panel report to the API Subcommittee on Corrosion last May.[2]

The API report points out that early corrosion experience with catalytic reformers indicated a new and severe type of attack. Later developments showed that metal loss from this type of attack was not as serious as anticipated and that the corrosion scale which interfered with continuous operation was the major difficulty. Today these problems are being dealt with primarily by feed desulfurization and by proper metal selection.

From a standpoint of metal selection, only the austenitic stainless steels appear to offer satisfactory resistance to high-temperature hydrogen sulfide corrosion under a wide range of conditions. Data reported by a number of investigators[2] bear out this general statement. This does not mean, however, that all high-temperature hydrogen sulfide corrosion problems can be solved most economically by using austenitic stainless steels. Under some conditions, even these metals corrode at a high enough rate to cause excessive scale formation. Recent data obtained in the authors' laboratory study shed more light on the behavior of stainless steels in high-temperature hydrogen-hydrogen sulfide atmospheres and therefore are presented in this paper.

Earlier work[1] shows that the conventional materials of construction may be divided into three general groups, with respect to their corrosion rate in a hydrogen-hydrogen sulfide atmosphere; namely, the 0-5 percent chromium steels, the 7-16 percent chromium steels, and the chromium-nickel steels. The austenitic chromium-nickel stainless steels provide the best corrosion resistance over a wide range of conditions. The group of carbon and low chromium steels exhibit the poorest corrosion resistance. The intermediate group of 7-16 percent chromium steels are unpredictable; they are usually no better than lower chromium steels except at mild conditions.

★ A paper presented at a meeting of Technical Group Committee T-8, Oklahoma City, Oklahoma, October 1, 1957.

(1) Socony Mobil Oil Company, Inc., Research and Development Laboratory, Paulsboro, New Jersey.

TABLE 1—Summary of Laboratory Corrosion Test Results on Stainless Steels

Run No.	L-1	L-2	L-3	L-4	L-5	L-6	L-7	L-8	L-9	L-10	L-11	L-12	L-13	L-14	L-15
Average Conditions															
Temperature, Degrees F	500	525	600	700	700	750	800	900	950	960	970	985	1120	1390	1390
Hydrogen Pressure, psig	465	465	465	465	465	595	465	465	175	485	485	485	485	185	185
H_2S Concentration in H_2, % Vol.	0.30	0.30	0.30	0.03	0.10	10.0	0.30	0.30	0.10	0.80	0.20	0.75	0.05	0.05	0.15
Length of Test, Hours	181	236	259	183	230	274	275	246	598	461	234	222	415	458	468
Materials Tested	Corrosion Rate, In./Yr.														
0-5% Cr Steels[1]	0.002	0.012	0.025	0.025	0.064	0.23	0.14	0.10	0.12	0.34	0.25	0.46	0.10	0.001	0.038
12 Cr Steel—Type 405	0.0004	—	0.022	0.002	—	—	0.11	0.050	0.075	—	—	—	—	0.013	0.18
12 Cr Steel—Type 410	0.004	—	—	0.003	0.053	—	0.11	—	—	0.22	0.19	0.30	0.10	—	0.16
16 Cr Steel—Type 430	0.0008	0.003	0.006	0.008	0.014	0.013	0.011	0.016	—	0.039	0.030	0.060	—	0.016	0.24
26 Cr Steel—Type 446	0.002	0.009	0.013	0.011	0.012	0.006	0.005	0.010	0.010	0.015	0.026	0.042	0.041	0.076	0.23
Cr—Ni Steels[1]	0.0001	0.001	0.001	0.002	0.003	0.007	0.003	0.007	0.007	0.023	0.027	0.040	0.012	0.005	0.065

[1] Test results are average of several measurements; others are individual measurements.

TABLE 2—Tests in Commercial Catalytic Reforming Unit

Estimated Average Conditions	1st Test	2nd Test
Temperature, Degrees F	1150	1110
Total Pressure, psig	190	185
H_2S Concentration, % Vol.	0.03	0.005
Exposure Time, Days	55	318
Materials	Corrosion Rate, In./Yr.	
0-9% Cr Steels	0.110	0.040
12-16% Cr Steels	0.065	0.030
Cr-Ni Steels	0.055	0.024

TABLE 3—Effect of Heat Treatment on Corrosion Rate of Stainless Steels

	Run No.: L-16 to L-18	
Average Conditions		
Temperature, Degrees F	750	
Hydrogen Pressure, psig	485	
H_2S Conc. in H_2, % Vol	1.5	
Length of Test, Hours	500–775	
Materials	Heat Treatment[1]	Corrosion Rate, In./Yr.
18-8S, Type 304 (Wrought)	None	0.0065
18-8S, Type 304 (Wrought)	A	0.0060
18-8S, Type 304 (Wrought)	C	0.0025
18-8SL, Type 304L (Wrought)	None	0.0052,0.0048
18-8SL, Type 304L (Wrought)	B	0.0019,0.0013
18-8SL, Type 304L (Wrought)	None	0.0057
18-8SL, Type 304L (Wrought)	A	0.0048
18-8SL, Type 304L (Wrought)	C	0.0070
18-8C, Type 347 (Wrought)	None	0.0102,0.0120
18-8C, Type 347 (Wrought)	B	0.0021,0.0016
18-8C, Type 347 (Wrought)	None	0.0072
18-8C, Type 347 (Wrought)	A	0.0043
18-8C, Type 347 (Wrought)	C	0.0076
18-8C, Type 347 (Weld)	None	0.0073
18-8C, Type 347 (Weld)	A	0.0051
18-8C, Type 347 (Weld)	C	0.0057
25-20, Type 310 (Wrought)	None	0.0041
25-20, Type 310 (Wrought)	A	0.0028
25-20, Type 310 (Wrought)	C	0.0030

[1] A—Heated 2 days at 1400 F, slowly furnace cooled.
B—Heated 2 hours at 1400 F, air quenched.
C—Heated 4 hours at 1650 F, rapidly cooled in air.

Two General Types of Stainless Steels

Included in the unpredictable 7-16 percent chromium steel group are straight-chromium stainless steels of the 400-series, which do not have an austenitic structure, as do the stainless steels of the 300-series. To compare these straight-chromium stainless steels with the other steels, a summary of corrosion rates is shown in Table 1. This summary includes all laboratory runs of more than 180 hours exposure that included both a 26 percent chromium steel and at least one other straight-chromium stainless steel. A wide range of conditions is shown; data appear for temperatures from 500 to 1400 F and for hydrogen sulfide concentrations of 0.03 to 10 percent volume. For comparison, Table 1 also shows the corrosion rates obtained on the 0-5 percent chromium steels and on the austenitic chromium-nickel stainless steels. Study of the table shows that, while the data are scattered, some conclusions may be drawn:

(1) For temperatures in the operating range of most catalytic reformers and naphtha desulfurizers, the austenitic stainless steels are the most resistant to hydrogen sulfide corrosion, and the carbon and low chromium steels are the least resistant. The straight-chromium stainless steels usually have a resistance somewhere between the other two groups.

(2) The corrosion rates of the 0-5 percent chromium steels increase with temperatures up to about 1000 F. Beyond this temperature the corrosion rates decrease. This decrease in corrosion rate can be predicted from Rosenquist's thermodynamic data, as described in a paper by Dravnieks and Samans.[3] The corrosion rates of the stainless steels also increase with temperature, but these rates continue to increase to at least 1400 F (the highest temperature at which the authors have data). As a result, the corrosion rates of the stainless steels become greater than those of the 0-5 percent chromium steels at some temperature between 1000 and 1400 F. It will be noted that at 1400 F none of these steels has sufficient corrosion resistance to hydrogen sulfide, for practical use, except at very low hydrogen sulfide concentrations.

(3) Below 1000 F the corrosion rates for the 12 percent chromium steels are generally not greatly lower than the rates for the 0-5 percent chromium steel, except at low hydrogen sulfide concentration. Increasing the chromium content of the steel to 16-26 percent will generally show a marked lowering of corrosion rate. Below 700 F the corrosion rate for the 26 percent chromium steel is higher than the rate for the 16 percent chromium steel; above 700 F the rate is slightly lower. At temperatures around 1000 F and at high hydrogen sulfide concentrations, there is no significant difference in corrosion rate for 16 percent chromium, 26 percent chromium, and the austenitic stainless steels. At 1400 F the straight-chromium stainless steels exhibit corrosion rates considerably higher than either the austenitic stainless steels or the 0-5 percent chromium steels.

Laboratory data below 1000 F show that the corrosion rates for the austenitic stainless steels are less than one-tenth the rates for the 0-5 percent chromium steels. As already pointed out, above 1000 F the corrosion rates for the austenitic stainless steels begin to approach the rates of the 0-5 percent chromium steels and may actually be greater at higher temperatures. These laboratory data are supported qualitatively by re-

TABLE 4—Comparison of Wrought, Weld, and Cast Stainless Steel Corrosion Rates

	Run L-19	Run L-20
Average Conditions		
Temperature, Degrees F	750	975
Hydrogen Pressure, psig	485	485
H_2S Conc. in H_2, % Vol.	1.5	1.5
Length of Test, Hours	590	990
Materials	Corrosion Rate, In./Yr.	
5 Cr—½ Mo	0.130	0.285
Type 304, Wrought	0.0122	0.0239
Type 304, Weld	0.0083	0.0171
Type 304, Cast	0.0084	0.0154
Type 304L, Wrought	0.0104	0.0250
Type 304L, Weld	0.0078	0.0212
Type 304L, Cast	0.0057	0.0123
Type 316, Wrought	0.0112	0.0208
Type 316, Cast	0.0113	0.0220
Type 316L, Wrought	0.0119	0.0240
Type 316L, Cast	0.0052	0.0117
Type 310, Wrought	0.0049	0.0145
Type 310, Weld	0.0047	0.0117
Type 310, Cast	0.0030	0.0123

TABLE 5—Chemical Analysis of Metals Used in Corrosion Test

Description	Supplier	Heat Number	C	Cr	Ni	Mo	Mn	P	S	Si	Al	Cu	Cb	Ta	N
Type 304—Wrought	G. O. Carlson Co.	64343-2B	0.055	18.56	9.42	—	0.67	0.030	0.025	0.44	—	0.25	—	—	—
Type 304—Weld (Type 308)	A. O. Smith Corp.	—	0.08	20.42	10.11	—	1.73	0.018	0.013	0.36	—	—	—	—	—
Type 304—Cast (CF8)	Alloy Steel Casting Co.	6498	0.041	19.33	10.33	—	1.49	—	—	1.13	—	—	—	—	—
Type 304L—Wrought	G. O. Carlson Co.	64541-2B	0.028	18.29	9.44	—	1.09	0.024	0.013	0.39	—	0.19	—	—	—
Type 304L—Weld (Type 308 ELC)	A. O. Smith Corp.	—	0.03	20.85	11.09	—	1.74	0.02	0.028	0.37	—	—	—	—	—
Type 304L—Cast (CF4)	Alloy Steel Products Co.	9074	0.03	18.65	9.80	—	0.63	—	—	0.94	—	—	—	—	—
Type 316—Wrought	G. O. Carlson Co.	64073-1C	0.041	17.78	12.93	2.42	1.84	0.023	0.017	0.70	—	0.20	—	—	—
Type 316—Cast	Alloy Steel Casting Co.	6552	0.035	17.65	11.47	2.88	1.22	—	—	1.06	—	—	—	—	—
Type 316L—Wrought	G. O. Carlson Co.	64307-2	0.019	17.60	13.80	2.75	1.50	0.021	0.019	0.55	—	0.34	—	—	—
Type 316L—Cast (CF4M)	Alloy Steel Products Co.	7053	0.019	19.32	10.49	2.20	0.52	—	—	0.83	—	—	—	—	—
Type 347—Wrought	G. O. Carlson Co.	54708-1A	0.060	18.69	11.29	—	1.65	0.022	0.012	0.68	—	0.23	0.69	0.13	—
Type 347 CBS—Weld	A. O. Smith Corp.	—	0.05	20.16	10.80	—	1.56	0.022	0.014	0.30	—	—	—	—	—
Type 309—Wrought	G. O. Carlson Co.	54580-24	0.060	22.24	14.62	—	1.93	0.020	0.014	0.46	—	0.17	—	—	—
Type 310—Wrought	G. O. Carlson Co.	54038-1B	0.060	24.76	20.07	—	1.25	0.021	0.015	0.55	—	0.12	—	—	—
Type 310 CBS—Weld	A. O. Smith Corp.	—	0.12	27.35	21.75	0.19	1.72	0.018	0.012	0.41	—	—	—	—	—
Type 310—Cast (HK)	Alloy Steel Casting Co.	6607	0.144	26.03	20.89	—	0.90	—	—	1.24	—	—	—	—	—
Type 202	Allegheny Ludlum Steel Corp.	69446	0.096	17.70	4.07	—	10.24	0.027	0.009	0.66	—	—	—	—	0.13
Type 204 ELC	Allegheny Ludlum Steel Corp.	25350	0.024	17.22	5.39	—	9.99	0.024	0.010	0.64	—	—	—	—	0.20
USS Tenelon	United States Steel Corp.	7X1077	0.06	18.07	0.52	—	15.20	0.022	0.008	0.10	—	—	—	—	0.51
Croloy 20-2 Al	Babcock & Wilcox	—	0.056	21.42	0.14	0.04	0.42	0.014	0.006	1.08	2.24	0.07	—	—	—
12 Cr—4 Al	U. S. Pipe & Foundry	—	0.074	12.26	—	—	0.91	—	—	0.95	3.82	—	—	—	—

TABLE 6—Summary of Corrosion Test Results for Manganese-Modified Austenitic Steels

	Laboratory Tests						Commercial Reforming Unit Tests[1]			
	L-21	L-22	L-23	L-24	L-25	L-26	C-1	C-2	C-3	C-4
Average Conditions[2]										
Temperature, Degrees F	750	750	790	810	805	1030	730	685	700	925
Pressure, psig	485	595	200	500	785	175	500	500	450	575
H_2S Conc. in H_2% Vol	1.5	10.5	10.2	6.2	11.8	0.10	0.01-0.1	0.01-0.1	0.05-0.15	0.001-0.01
Materials Tested[3]	Corrosion Rate, In./Yr.									
0—5% Cr Steels	—	0.23	0.29	—	0.33	0.16	0.059	0.033	0.017	0.027
Cr-Ni Steels	0.0060	0.007	0.025	0.026	0.029	0.010	0.0005	0.0011	0.0010	0.0023
18 Cr—9 Mn—5 Ni, Type 202	0.0077	—	—	—	—	0.026	0.0005	0.0011	0.0009	0.0035
18 Cr—9 Mn—6 Ni, Type 204 ELC	0.0050	0.010	0.016	0.015	0.025	—	0.0004	0.0011	0.0009	0.0045
18 Cr—15 Mn—½ N, USS Tenelon	0.0042	0.011	0.010	0.009	0.011	—	0.0004	0.0020	0.0015	0.0064

[1] Each column represents the average of three tests at similar locations.
[2] The exposure time was 12-30 days for laboratory tests and 100-200 days for the commercial tests.
[3] Results for the 0-5 percent Cr and Cr-Ni steels are average of several measurements; the manganese-modified steels are single measurements for each test.

sults on test coupons placed in the recycle heater transfer line of a Thermofor Catalytic Reformer,[4] as shown in Table 2. The commercial unit data at 1100 F show that the corrosion rate for the austenitic stainless steels is only one-half the rate for the 0-9 percent chromium steels.

Heat Treated Stainless Steels

During fabrication of stainless steel components for commercial use, it may be necessary, or even advisable, to include a heat treatment step. Therefore, it is important to know what effect various heat treatments may have on corrosion rates. Before subjecting various wrought test coupons of the 300-series to a hydrogen-hydrogen sulfide atmosphere in laboratory units, several coupons were heat-treated, as described in Table 3. Heat treats A and B are sensitizing treatments which produce intergranular precipitation of carbides. These laboratory procedures should reproduce a condition which may exist as a result of fabrication and welding. Heat treat C is a stabilization treatment used at times to overcome a tendency toward intergranular attack and cracking.

In almost all cases the heat treatment caused a significant reduction in corrosion rate of wrought specimens. The same trend also was observed for Type 347 weld metal.

Wrought, Weld and Cast Stainless Steels

Most of the high-temperature hydrogen sulfide corrosion data in the literature are reported for wrought steels. However, integral parts of a commercial unit may also require the use of cast materials and, of course, a number of different welding rod compositions may be employed. Therefore, it becomes important to know the relative corrosion rates for wrought, weld, and cast stainless steels in a hydrogen-hydrogen sulfide atmosphere.

Laboratory tests have been made on many of the conventional austenitic stainless steels; namely, Types 304, 304L, 316, 316L, and 310, as reported in Table 4. The results show that both weld and cast stainless steels exhibited lower corrosion rates than wrought metal. The chemical compositions of the weld and cast metals tested are reported in Table 5, along with the compositions of the other stainless steels reported in this paper.

Manganese-Modified Austenitic Stainless Steels

In recent years, while nickel was in critical supply, considerable attention was paid to the new manganese-modified austenitic stainless steels. Because of this interest, several of these new stainless steels have been exposed to high-temperature, hydrogen-hydrogen sulfide streams, both in laboratory and commercial units. The data obtained are summarized in Table 6.

These results show that the corrosion rates for the manganese-modified steels are of the same order as for the conventional austenitic stainless steels below 900 F. Above this temperature, these new steels may exhibit somewhat higher corrosion rates than the conventional steels. In general, the commercial unit tests confirm those conducted in the laboratory.

It will be noted that the corrosion rate for USS Tenelon was lower than the rates for the Type 202 and Type 204 ELC steels in laboratory tests; however, in the commercial unit, USS Tenelon exhibited a slightly higher rate than did the other manganese-modified steels. The reason for this difference is unknown.

Miscellaneous Metals

Certain materials not commonly used for high temperature hydrogen sulfide corrosion protection also were tested. These include high-nickel-chromium al-

loys and certain high chromium-aluminum steels. These materials were tested at 750 F and 1.5 percent volume hydrogen sulfide. Their corrosion rates may be compared with rates for austenitic stainless steels in Table 7.

The high-nickel-chromium alloys are of interest because of their resistance to stress corrosion cracking. Three such alloys tested exhibited corrosion rates of the same magnitude as the austenitic stainless steels, Incoloy being somewhat lower and Inconel slightly higher. This same relative trend for Incoloy, Inco 804, and Inconel was found in another test at somewhat different conditions (700 F, 465 psig, 0.3 percent vol. hydrogen sulfide in hydrogen for 185 hours). These materials, however, have not been investigated at higher temperatures.

Aluminum is very resistant to high temperature hydrogen sulfide attack, but its poor mechanical properties preclude its use in pressure equipment. For the test reported (Table 7) commercial aluminum showed no metal loss. German literature of the early 1930's indicates that aluminum addition to steels, particularly 5 percent or more, may improve the high-temperature hydrogen sulfide corrosion resistance of the metal. For this reason aluminum and chromium-aluminum steels were included in the test reported in Table 7. A special alloy, obtained from the U. S. Pipe and Foundry Metallurgical Research Department, containing 12 percent chromium and 4 percent aluminum also showed no metal loss. A similar alloy, Croloy 20-2Al, exhibited a low corrosion rate of 0.003 in./yr. at these test conditions. Croloy 20-2Al, however, exhibited corrosion rates 50 to 100 percent greater than the austenitic stainless steels at higher temperatures of 1000-1500 F.

Stress Corrosion Cracking

As has been indicated above, the use of austenitic stainless steels is often required because of their superior resistance to high-temperature, hydrogen-hydrogen sulfide corrosion. These steels are, however, peculiarly susceptible to stress corrosion cracking in certain environments that may exist in commercial units such as reformers. Apart from chlorides, which are well known to promote stress corrosion cracking of austenitic stainless steels, the literature reports[2,5] that polythionic acids formed by the action of moisture on iron sulfide can also have this effect.

The authors know of no case of stress corrosion cracking of stainless steel to have occurred because of high-temperature, hydrogen-hydrogen sulfide corrosion during the actual operation of a commercial unit. Stress corrosion cracking failures have occurred during down periods, and it is thought that polythionic acids formed, as explained above, are the cause. Current practice in the field is to avoid the formation of polythionic acids during shutdowns, or to neutralize acids before any harm is done, as described by E. L. Hildebrand.[5]

A preliminary attempt has been made to reproduce this stress corrosion cracking phenomenon in the laboratory. This was done by exposing stressed specimens first to a high temperature sulfide atmosphere long enough to build up a scale, and then to a condensing steam atmosphere.

Figure 1 shows the actual test set-up. The 1½ x ½ x ¼ or ⅛ inch specimens

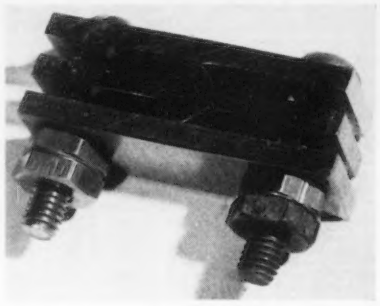

Figure 1—Photograph of stressed specimen couple.

were tested in pairs. As shown in the figure, a fulcrum was placed at the center between two identical specimens, and the ends of the specimens were pulled together by bolts. By carefully measuring the deflection of the ends of the specimens after tightening the bolts, it was possible to calculate the stresses in the top fibers of the specimens.

In the initial test, specimens stressed at 10,000 psi were subjected to sulfidizing conditions (700 F, 485 psig, 1.5 percent vol. hydrogen sulfide in hydrogen) for 48 hours, followed by exposure to condensing steam for four hours. All the conventional austenitic steels were tested in this manner. No cracking was observed; therefore, the same specimens were stressed above the yield point (i.e., above 30,000 psi) and returned to the sulfidizing atmosphere for an additional 94 hours. After four hours of steaming, only one specimen, Type 304, showed visible cracking. A photograph of this cracked specimen is shown in Figure 2. None of the other coupons showed microscopic cracking on metallurgical examination.

Further investigation revealed that the cracked Type 304 specimen had been severely cold-worked prior to the test cycle. Therefore, when similar preliminary tests were performed using wrought specimens having an area of weld metal in the center, several of these specimens were marked with a chisel to give areas of cold-worked metal. The exploratory tests included all conditions expected in the fabrication of a unit. Tests on artificially sensitized material were not included; however, the weld area should have been sensitized to some degree in the Type 304 welded specimens. No failure of any of these specimens, attributable to stress corrosion cracking by polythionic acids, was observed after stressing, sulfidizing, and steaming.

The authors do not consider these preliminary tests to be conclusive and additional work is needed to determine the role of polythionic acids on stress corrosion cracking of stainless steels.

Conclusions

The corrosion results for stainless steels in high-temperature, hydrogen-hydrogen sulfide atmospheres may be summarized as follows:

1. Under conditions that are most likely to be encountered in commercial catalytic reforming and desulfurization units, the 300-series austenitic stainless steels afford better resistance to high-temperature hydrogen sulfide corrosion than do the straight-chromium stainless steels of the 400-series.

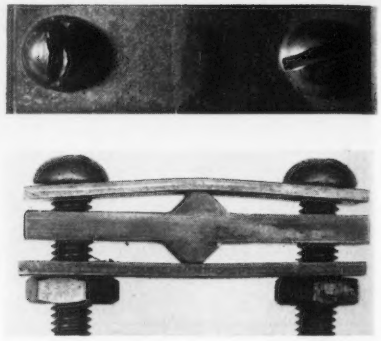

Figure 2—Two views of Type 304 specimen that cracked in laboratory test. Note failure of upper specimen due to crack in middle of specimen (visible in top view).

TABLE 7—Corrosion Test Results for Miscellaneous Metals

	Run L-21
Average Conditions	
Temperature, Degrees F	750
Hydrogen Pressure, psig	485
H_2S Conc. in H_2, % Vol	1.5
Length of Test, Hours	730
Materials	**Corrosion Rate, In./Yr.**
18-8S—Type 304	0.0079
18-8C—Type 347	0.0065
25-12S—Type 309	0.0049
18 Cr—9 Mn—5 Ni—Type 202	0.0077
18 Cr—9 Mn—6 Ni—Type 204 ELC	0.0050
18 Cr—15 Mn—½ N—USS Tenelon	0.0042
34 Ni—21 Cr—Incoloy	0.0029
42 Ni—30 Cr—Inco 804	0.0050
78 Ni—15 Cr—Inconel	0.0101
Commercial Aluminum	Nil (untarnished)
12 Cr—4 Al—Special Alloy	Nil (slight tarnish)
20 Cr—2 Al—Croloy 20-2Al	0.0030

2. The manganese-modified and the conventional austenitic stainless steels exhibited corrosion rates of the same order, both in laboratory and commercial unit tests.
3. Heat treating austenitic steels at 1400-1650 F for from two hours to two days, followed by air quenching or slow furnace cooling, improved the resistance of these steels to high-temperature hydrogen sulfide corrosion.
4. Corrosion rates for wrought stainless steels are higher than for weld or cast steels of similar compositions.
5. The authors' preliminary tests on stress corrosion cracking are inconclusive and additional information will be needed to understand the role moist iron sulfide scale plays in promoting cracking of austenitic steel.

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Breakaway Oxidation of Zirconium-Tin Alloys*

By EARL A. GULBRANSEN* and KENNETH F. ANDREW

Abstract

A breakaway oxidation reaction is found for zirconium-tin alloys similar to the breakaway corrosion reaction found for steam reactions. As oxidation is a simple reaction to study, the basic breakaway phenomena may be elucidated.

Data are given to show the breakaway oxidation of Zircaloy II at temperatures from 600 C to 800 C. 3.2.3

Introduction

BREAKAWAY CORROSION reactions have been observed for both zirconium and zirconium-tin alloys in air and oxygen as well as in steam atmospheres.[1,2,3] In steam, Thomas[3] has observed for a 1.8 percent alloy containing small amounts of iron, nickel, and chromium that breakaway corrosion at 400 C occurred at a weight gain of 350.0 micrograms per sq cm for both 1 and 100 atmospheres pressures.

Kendall[1] in a study of the air oxidation of Zircaloy II containing 1.5 percent Sn showed that breakdown occurred at a weight gain of 1000 micrograms per sq cm at 600 C and 600 micrograms per sq cm at 500 C, the times for breakdown being nine hours at 600 C and 40 hours at 500 C. From the shape of Kendall's 700 C experiment it would appear that breakaway reaction occurs for a reaction time of less than two hours.

In contrast to Kendall's results Mallett and Albrecht[2] have observed on rod specimens that the time for breakdown reaches a minimum value of about 30 hours at 700 C and increases to 100 hours at 800 C. This unusual behavior can be explained only by assuming that the weight gain for breakdown increases rapidly with temperature. In addition the shape factor of the specimen may play an important role. The work of Garibotti, Green, and Baldwin[4] also should be mentioned since these authors have shown that both oxygen and nitrogen must be present to give the high oxidation rates observed in air for the pure metal.

This note will present evidence on the time, temperature, and weight gain conditions for breakaway oxidation on Zircaloy II containing 1.45 percent Sn and small amounts of Fe, Ni, and Cr.

Experimental Procedure

A vacuum microbalance and associated apparatus[5,6,7] was used to study the kinetics of the oxidation process. Samples of the alloy were obtained from the Westinghouse Atomic Power Division. The alloy has the following analyses:

Sn	1.45
Fe	0.135
Ni	0.055
Cr	0.100
N	0.0038
H	0.0020
O	0.1400
Al	0.0045
Hf	0.0055

The sample surface was prepared by abrading through 4/0 paper under purified kerosene and cleaned.[5] The specimens had surface areas of 1.4 sq cm and weighed 0.1235 gm.

Each of the samples was reacted with 0.1 atmosphere and oxygen in a manner previously described.[5,6,7,8] Figure 1 shows five oxidation experiments for temperatures of 600 C to 800 C. Here the weight gain in units of micrograms per sq cm is plotted against the time in minutes. The onset of a change in oxidation mechanism is indicated by the arrows for each temperature.

The weight gain at which breakaway oxidation is observed depends upon the time and temperature. At 600 C and 650 C the weight gain for breakaway is about 1000 micrograms per sq cm while the weight gain at 750 C is 1650 micrograms per sq cm. The times for transition in the oxidation rate is nine hours at 600 C and two hours at 750 C.

Curve A in Figure 1 for the 800 C run indicates that breakaway conditions have not been reached. However, a parabolic rate law plot of the data shows that a breakaway condition is beginning at 2750 micrograms per sq cm. Since the oxidation rate can be fitted to the parabolic rate law, the change in oxidation mechanism is best shown in the parabolic rate law plots. Figure 2 shows a plot of the weight gain squared as a function of time for the 700 C experiment.

The effect of temperature on the weight gain at which breakaway oxidation begins is shown in Figure 3. The authors' explanation of the rapid increase of the weight gain for breakdown is that the solution of oxide in the alloy is becoming of increasing importance for the higher temperature experiments. The thickness of the oxide film for breakaway oxidation as a result is not that given by the measured weight gain.

Although breakaway corrosion reactions have been observed for zirconium and its alloys, similar effects are observed on other metals.[9] In fact all but the noble metals fail in corrosion reactions for one or more reasons.

Conclusions

It is concluded that the conditions for breakaway oxidation of a zirconium-tin alloy are a function of time and temperature. The weight gain for breakaway oxidation increases with the reaction temperature and is probably due to the solution of oxygen in the alloy. The rapid increase in the weight gain for breakaway with temperature explains in part the results of Mallett and Albrecht and the fact that a minimum time for breakaway oxidation is found in Figure 1.

Since the oxidation reaction can be studied under more controlled conditions, it may be possible to elucidate the fundamental factors underlying this interesting phenomena, and thus improve the performance of a particular material.

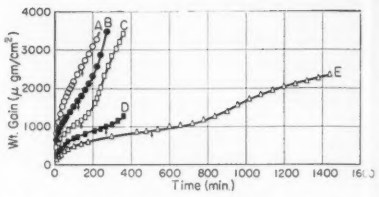

Figure 1—Breakaway oxidation of Zircaloy II for temperatures ranging from 600 C to 800 C with 7.6 cm of Hg or O_2. Metal was abraded through 4/0 paper. Code to curves is as follows: A—800 C, B—750 C, C—700 C, D—650 C, E—600 C.

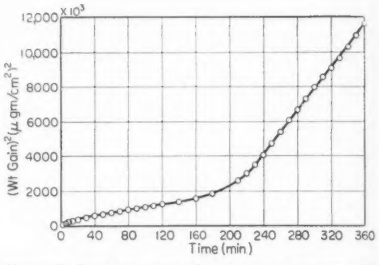

Figure 2—Parabolic plot of oxidation of Zircaloy II at 700 C and 7.6 cm of Hg of O_2.

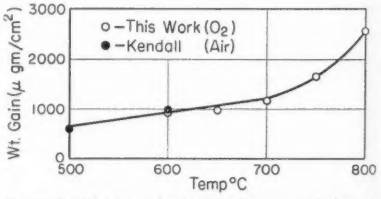

Figure 3—Weight gain for breakaway oxidation as a function of temperature for Zircaloy II.

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★ Submitted for publication January 9, 1957.

* Westinghouse Research Laboratories, Pittsburgh, Pennsylvania.

Publication 58-4

NACE TECHNICAL COMMITTEE REPORT

Theoretical Aspects of Corrosion in Low Water Producing Sweet Oil Wells

A Status Report of NACE Technical Unit Committee T-1C,[1]
Prepared by Task Group T-1C-4[2]

Abstract

A study was conducted which confirmed that the principal corrosive agents in low water producing sweet oil wells are hydrolytic products of inorganic salts. These salts may be deposited on metal either in the form of a thin layer or as a consolidated scale. In addition to hydrolysis of the salts, electrochemical cells are set up between the metal and the various scale constituents and thus add to the corrosion.

A mechanism is discussed for scale deposition. Inorganic salts are carried from the producing formations by water globulae and suspended paraffin. Where conditions are favorable, the carrying media separate from the crude and lay down as loose layer. The deposition continues until a consolidated, aggregate scale results.

Physical appearance of the scale and changes in physical conditions were found to affect the corrosion rate.

Conventional criteria were found to be of little value in detecting corrosion in low producing sweet wells in which scale was deposited. It was thought that corrosion could be detected in such cases through analyses of fluids for acid emulsions, inorganic salts and hydrocarbon mass. 8.4.3

Introduction

PREVIOUS REPORTS by Committee T-1C and Task Group T-1C-4, based on work done by the chairman, have shown that hydrolysis of sulfate scale may be the reason for corrosion in low water[3] producing sweet oil wells (previously called water independent corrosion). Data from the NACE questionnaire concerning corrosion and scale formation in the low water producing sweet oil wells indicate that this type of corrosion is a serious, but not a widely spread, problem. Of the 2,613 low water producing sweet oil wells considered, about 8 percent suffered severe corrosion. These wells were located along the Louisiana Gulf Coast, Texas Gulf Coast and in Venezuela.

The service life of tubing in corrosive wells is about 3-4 years. A characteristic feature of corrosion which has been detected on the pulled tubing is localized pitting underneath the scale. One company also has reported pitting underneath the paraffin deposit. The scale which has been found in these corrosive wells is composed of $CaSO_4$, $CaCO_3$, NaCl, $MgCl_2$, $FeCl_2$ and iron oxides.

The pitting type attack has been reproduced successfully under laboratory conditions and the results reported. The mechanism of corrosion occurring after the scale has been deposited is understood. There is, however, lack of simple criteria to predict corrosion and to clarify the occurrence of scale constituents before they appear on the tubing. A study was conducted therefore on the occurrence and properties of scale-forming components.

The study confirmed that the principal corrosive agents in the low water producing sweet oil wells are hydrolytic products of inorganic salts. These salts cause corrosion when deposited on metal either in the form of a thin layer or as a consolidated scale. The frame of the consolidated scale is composed of less soluble sulfates which enclose more corrosive and soluble chlorides. The hygroscopic chlorides tend to extract water from the crude and through hydrolysis of the salts cause a low pH in and underneath the scale.

In addition to hydrolysis of the salts, electrochemical cells are set up between the metal and the various scale constituents. These cells add to the corrosion; however, their effect cannot be distinguished from the effect of hydrolysis. Moreover, carbon dioxide and organic acids contribute slightly to the corrosion rate.

Hydrolysis is reversal of neutralization. Hydrolytic products are formed when a salt composed of an acid and a base, each of different strength, is combined with water. The resulting solution shows either acid or alkaline properties. Most of the inorganic salts which occur in the low water producing sweet oil wells are composed of strong acids and weaker bases; when they are combined with water they have a tendency to give off hydrogen ions. The hydrolytic constants

(1) Joy T. Payton, The Texas Co., Houston, Texas, chairman.
(2) Rado Loncaric, Atlantic Refining Co., Dallas, Texas, chairman.
(3) Wells producing from trace to 3 percent water.

Figure 1

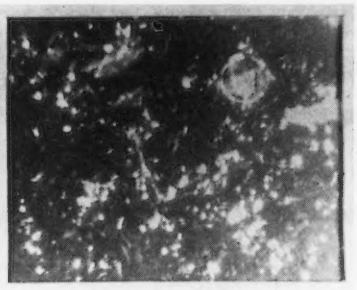

Figure 2

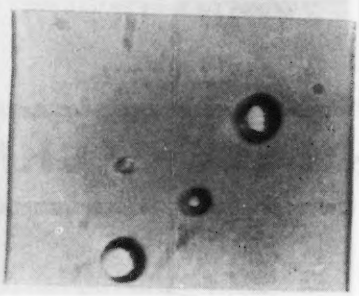

Figure 3

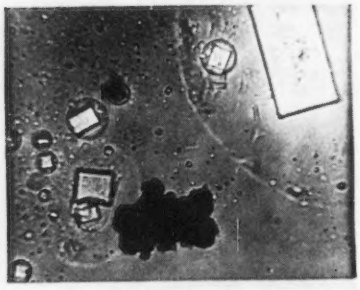

Figure 4

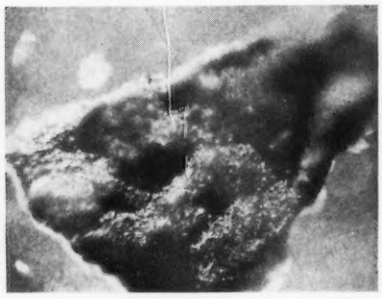

Figure 5

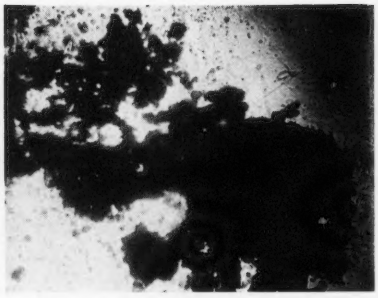

Figure 6

which are derived from the disassociation constants of the bases and the ionic product of the water show that pH values from 3-4 can be obtained through hydrolysis of the inorganic salts found in the low water producing sweet oil well. The hydrolytic products of such bases as calcium, magnesium, and iron are less soluble in the small amount of water which is produced in these wells, than are the hydrolytic products, such as chlorides and sulfates. Thus, these acids are set free and can cause up to 58 mpy penetration in metal.

In the crude, inorganic salts are enclosed in water globulae and embedded in paraffin. The minute water globulae (about 5-7 microns in size) are surrounded by an oil film, Figure 1, to which paraffin particles (1-2 microns) are attached, Figure 2. These paraffin particles are emulsion stabilizers and clot easily at increased temperature. Most of the water globulae contain inorganic nuclei, Figure 3, which grow in size where the water is allowed to evaporate, Figure 4. The pH value in the water globulae ranges from 3-4.

The paraffin (a mixture of aliphatic and aromatic hydrocarbons, asphalts, resins and naphthenes) occurs in the crude in the form of suspension, Figure 2. The size of particles ranges widely (1-100 microns). The composition of paraffin varies from field to field. It is the high melting paraffin that plays an important part in this type of corrosion. This paraffin, a mixture itself, encloses an appreciable amount of inorganic salts and water. A chemical analysis of high melting paraffin from one field shows organic compounds 64.1 percent; NaCl 4.1 percent; ferrous and ferric compounds (i.e. oxides, chlorides) 18.2 percent; $CaSO_4$ 11.1 percent; metallic iron 1 percent; water 1 percent. The paraffin particles have a tendency to grow and to form a dense dark hydrocarbon mass with a large surface area, Figure 5. The hydrocarbon mass is magnetic and can be extracted from the crude by a magnet.

The part which carbon dioxide and organic acids play in corrosion is understood and will not be specially discussed here. It should be noted that most corrosive wells in this category are high pressure wells, and carbon dioxide contributes to corrosion according to its partial pressure and temperature.

Mechanism of Scale Deposition

The mechanism of scale deposition appears to be complex. The inorganic salts are carried from the producing formations by the water globulae and suspended paraffin. These salts already have undergone some hydrolysis causing a low pH in both carrying media. Where conditions are favorable, the carrying media separate from the crude and lay down as loose layer. The deposition continues until a consolidated, aggregate scale results. The composition of the scale and its corrosive properties depend on the amount and physical properties of the impurities in the crude. If a crude contains calcium sulfate and a small amount of chlorides, the scale is composed mostly of sulfate. This scale consolidates easily and encloses the chlorides. The amount of chlorides determines the initial corrosion rate. A porous calcium sulfate scale without chlorides is less corrosive from the beginning because it has fairly high pH (about 6). However, the formation of the first corrosion product, ferrous sulfate, causes the pH to drop. In this case the corrosion product accelerates the corrosion rate. If the crude contains calcium sulfate and carbonate, the resulting scale consists of these two salts. Such a scale is less corrosive because of interaction between the acid sulfate and basic carbonate.

The degree of corrosion depends also on the physical appearance of the scale. A porous scale allows water globulae to penetrate it. This water in combination with salts causes corrosion through hydrolysis underneath the scale. Nonporous scale does not permit water to pass through it and therefore is not corrosive. Exactly what controls the porosity of scale is not known.

The consolidation of scale may be disturbed by a change of physical conditions, such as increased fluid velocity in the string, increased water production, sand erosion, killing the well with water, etc. If such disturbance occurs, the loose deposits are washed off the tubing, leaving the bare rough tubing surface reexposed to corrosive agents. Porous scale is more affected by a change of well conditions than is non-porous scale. If porous scale contains water soluble chlorides, they are likely to be extracted in case of increased water production. That should leave the less soluble and less corrosive sulfate in place.

The precipitation of high melting paraffin occurs when its weight or magnetism overcomes its floating ability. The corrosion underneath the layer results because of embedded inorganic salts and enclosed water. (Figure 6 shows the hydrocarbon mass on a coupon; Figure 7 shows the pit which resulted). Since the paraffin layer does not consolidate, it can be removed more easily than consolidated inorganic scale.

Some wells produce only traces of water and some up to 3 percent. When only traces occur, the water forms a uniform emulsion. When more than traces are produced, the water occurs in form of emulsions and as "free water." This free water tends to separate from crude "on standing." The amount of free

water produced by a well fluctuates. The effect of this water on corrosion is unknown.

Analyses of crudes indicate that over 2000 mg/1 of inorganic salts can be extracted from the corrosive crudes and about 20 times less from non-corrosive crudes. It was also noted that upon heating the corrosive crudes to 155-160 F, the oil film which surrounds water globulae breaks, and most water separates from the crude (see Figure 8). The cooled oil contains low melting paraffin and very few inorganic salts. The amount of these salts equals the quantity obtained from pipe line oil and non-corrosive power oil. Also the amount of the hydrocarbon mass in corrosive crudes was found to be higher than that in non-corrosive crudes. The total amount of paraffin in both crudes was the same.

In low water producing sweet oil wells in which scale was deposited, conventional criteria to detect corrosion are of little value for the following reasons:

1. The iron is retained by the scale and cannot be found in produced fluids
2. Caliper surveys indicate elevations and cavities of the scale rather than pits of the pipe if the scale is consolidated. If the scale is loose, caliper surveys are usable.

Figure 7

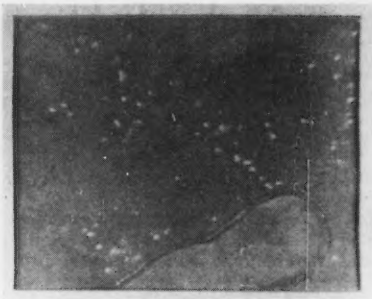

Figure 8

To detect corrosion in these cases, it is suggested that analyses of the fluids be made for acid emulsions, inorganic salts, and hydrocarbon mass. Best results should be obtained if wellhead fluid, as well as bottom hole fluid, is analyzed and the results compared. These results and the consideration of the bicarbonate—carbonate equilibria in a well should give a general picture of the well conditions.

To combat this type of corrosion very few practical measures are available at the present. The organic inhibitors are of little value because they seldom penetrate the scale to the tubing where protection is needed. It appears that a coating, because of its smooth surface, offers the least opportunity for inorganic salts to settle and form scale.

Fuel Ash Attack on Aluminum Coated Stainless Steel*

By J. E. SRAWLEY

Abstract

A laboratory investigation of the resistance of hot-dip aluminum coated Type 310 stainless steel to attack by residual oil ash constituents was carried out. The coated specimens did not exhibit better resistance than the uncoated steel, nor did prior heat-treatment of the coated specimens have an appreciable effect. The results of a boiler test were in agreement with the laboratory finding.
4.2.3

Introduction

IN RECENT YEARS the problem of attack by the combination of the gaseous products of combustion and the ash of residual fuel oils on the components of boilers and turbines has become one of the most serious limiting factors in the further development of shipborne power plants. Established materials deteriorate rapidly as temperatures are increased for the purpose of increasing efficiency. Presumably because of the complexity of the situation, no thoroughly satisfactory account of the conditions which may occur in boilers and turbines has yet been published. Most attention usually is given to the influence of vanadium compounds which are present in significant amounts in most residual oils. However, it has been emphasized[1,2] that the effects of compounds of sulfur, chlorine and sodium may be equally important. Reviews of published work on residual fuel oil ash attack, with adequate lists of references, have appeared recently in *Corrosion*.[3,4]

As a contribution to the widespread search for materials offering promise of superior behavior, an investigation was carried out at the U. S. Naval Research Laboratory on hot dipped aluminum coatings applied to Type 310 stainless steel. This is the composition commonly used for boiler and tube support plates. The undoubted value of aluminum coatings on steels in other applications[5] was justification for the investigation. Quite recently, the excellent promise of protection by aluminum in catalytic reforming and desulfurizing service in the petrochemical industry has been reported in *Corrosion*.[6]

Although the results of this investigation are unquestionably negative it is considered desirable that they should be available since there is, at this time, no explicit reference in the literature to the use of aluminum coatings in this type of application. Because the results are negative they are presented here in abbreviated form rather than in the detail of the U. S. Naval Research Laboratory Report.[7]

Experimental Procedure

Coatings were applied in the laboratory by dipping specimens cut from a single bar of commercial Type 310 stainless steel into a bath of 99.997 grade aluminum at 1400 F. A flux cover was used on the bath to ensure satisfactory coating of the whole surface of each specimen. After dipping, air-cooling and washing to remove adhering flux, some specimens were heat-treated to diffuse aluminum into the steel. Heat-treatment procedures will be identified in the discussion of results. After 24 hours at the testing temperature of 1700 F, diffusion had occurred to a sufficient extent that the effect of prior heat-treatment on the coating structure could hardly be distinguished. At this stage the average coating thickness was about 0.005 inch.

Testing consisted of heating specimens in an unsealed muffle furnace for periods up to 14 days. No attempt was made to control the atmosphere which therefore consisted of the ambient air at atmospheric pressure. The specimens were contaminated with a mixture of equal parts of sodium sulfate and vanadium pentoxide(1), the quantity of the contaminant being small in relation to the size of the specimen. In the least severe case a single initial application of one milligram per square centimeter of specimen surface was made, while in the severest case specimens were treated each day with four milligrams per square centimeter. Evaluation of the effect of corrodent contamination was carried out by microscopical examination of sections of the specimens normal to their surface. Specimens were usually cooled and weighed daily as an auxiliary means of following the progress of attack.

Results

Under the given experimental conditions the aluminum coatings did not reduce or delay attack on the specimens to any significant extent. Prior heat-treatment to diffuse the coatings did not influence the result. Figures 1 and 2 summarize the results of one illustrative experiment in which coated specimens of two types (the difference being in the prior heat-treatment) were compared with uncoated specimens. One heat-treatment consisted of heating to 1700 F in air, holding at temperature for one hour and then air-cooling. This was simply a nominal stabilizing treatment. The other heat-treatment was a proprietary process aimed at developing a special type of surface layer (a so-called preconditioning treatment). However, as will be seen, it was ineffective for the present purpose.

Specimens were contaminated with two milligrams per square centimeter of 50 percent Na_2SO_4: 50 percent V_2O_5, heated to 1700 F, held at temperature for 22 hours, cooled and weighed. This cycle was repeated for four days, specimens being removed after each cycle for microexamination. After the four days, the remaining specimens were once more contaminated and then remained at temperature for a further four days. Thus these specimens were at temperature for a total of eight days and were contaminated with a total of ten milligrams per square centimeter of corrodent mixture.

Figure 1 shows diagramatically the changes taking place at the surface of

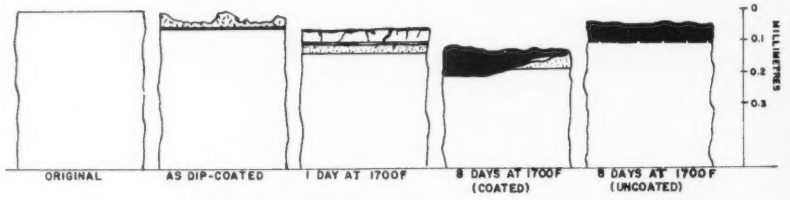

Figure 1—Sections of specimens before and after testing at 1700 F while contaminated with 50 percent Na_2SO_4 : 50 percent V_2O_5. Scale represents distance from original surface of steel.

(1) This contaminant was standardized after preliminary experiments in which various corrodent mixtures were used. These were either mixtures of sodium sulfate and vanadium pentoxide, or of these compounds with carbonaceous matter.[7]

★ Submitted for publication October 10, 1956.

About the Author

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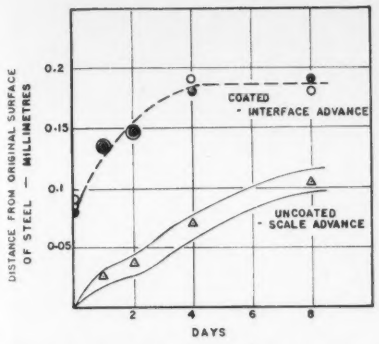

Figure 2—Advance of scale/metal interface of uncoated specimens and coating/parent-metal interface of coated specimens. Specimens were contaminated with 50 percent Na_2SO_4 : 50 percent V_2O_5 and exposed at 1700 F. Local penetration of coatings by scale occurred within eight days.

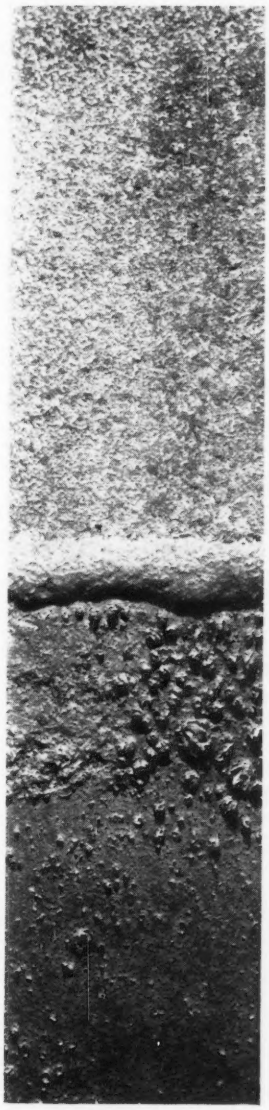

Figure 3—Appearance of composite plate after exposure in boiler.

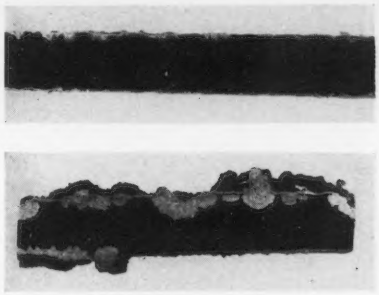

Figure 4—Macrosections through thickness of uncoated (top) and coated (bottom) parts of plate.

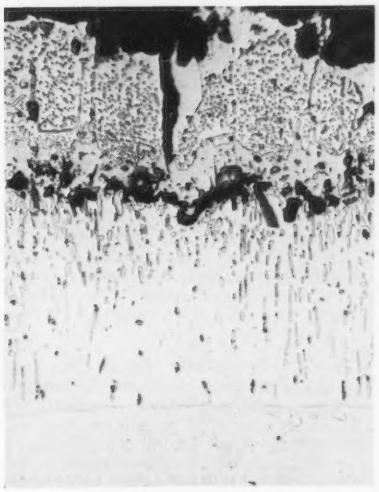

Figure 5—Structure of coating exposed several days at 1700 F but not severely attacked. Original magnification 500X; actual magnification after reduction for engraving purposes, 280X. Etched.

the specimens as determined from microexamination and measurement. The important feature is the comparison of the coated and uncoated specimens which had been exposed for eight days (there was no substantial difference between the two types of coated specimens). Although the penetration of scale into the coated specimen was irregular compared with the uncoated one, it had advanced beyond the coating/parent-metal interface in several places. This interface in turn had advanced well beyond the corresponding position of the scale interface of the uncoated specimen, as shown in Figure 1. Thus attack on the uncoated specimens had not reached a depth equal to that of the coatings by the time the coatings had been effectively penetrated. Under such circumstances it cannot be said that the coatings had any protective value.

In Figure 2 the results of microscopic measurement on all the specimens are plotted. The regularity of the attack on the uncoated specimens is shown in the lower curves representing the range of ten measurements per specimen. The upper curve represents the advance of the coating/parent-metal interface for the coated specimens. The same curve represents both heat-treatments within the experimental error, as shown by the plotted points. It is notable that the advance of the scale on the uncoated specimens was always behind the advance of the coating-metal interface. After eight days, when the coating had been penetrated, the scale front on the coated specimens in the penetrated regions was ahead of that on the uncoated specimens. These penetrated regions spread rapidly with increased exposure.

In other experiments heat-treatment of the coating for five hours at 1900 F was found not to improve the behavior, although the coating was much more effectively diffused by this heat-treatment than by those mentioned above. Coatings which were tested without any prior heat-treatment behaved as well as those that were heat-treated. This is not surprising in view of the fact that test (and service) conditions involve long exposure at temperatures where diffusion is quite rapid so that any heat-treatment may be regarded as the equivalent of a preliminary service exposure as far as diffusion is concerned.

A check on these results was provided by a test which corresponded closely to one type of service. This example was provided by the U. S. Naval Boiler and Turbine Laboratory. One-half of a piece of stainless steel sheet was dip-coated with aluminum and then joined to the other uncoated half by butt welding. The composite piece was then exposed in a boiler. Figure 3 shows the appearance of the composite piece after removal from the boiler while Figure 4 shows macro-sections through the sheets. The sections were photographed by oblique illumination so that the metal appears dark and the corroded regions relatively bright. Quite clearly the coated material has deteriorated to a greater extent than the uncoated steel. Microexamination revealed only traces of the original coating between the areas of deepest attack.

Microstructures

The structure of a relatively undamaged coating, such as would be obtained by heating the dipped specimen in air alone for several days at 1700 F or several hours at 1900 F, is shown in Figure 5. Temperature and duration of heating affect only the thickness of the layers and not the other features of the structure provided that: (1) Structure has formed, and (2) Depletion of aluminum by oxidation at the surface was not great enough to have an effect. There are three characteristic layers: (1) the outer, two-phase layer, (2) a narrow intermediate single-phase layer and, (3) an inner two-phase layer which has a sharp boundary with the parent steel. Microhardness measurements indicate that the aluminum content beyond this boundary is low, probably less than 1 percent. Examination of psuedo-binary alloys of Type 310 composition with aluminum indicates the solubility of aluminum in austenite of this composition to be of the order of 1 percent or less.

The two phases in the outer and the inner duplex layers are apparently the same, although almost certainly of different composition. As aluminum is depleted by continued oxidation at the surface the relative amounts of the two phases in the outer layer change and the intermediate single-phase layer diminishes. At the same time the inner, duplex layer advances into the steel.

Figure 6, at lower magnification, shows penetration of the coating in one of the 8-day specimens referred to ear-

lier (Figures 1 and 2). The residual coating appears at the right-hand side of the picture and close examination shows it to be depleted of aluminum compared with a coating heated in air alone for the same period.

Conclusion

It is evident from these results, which are supported by others[7], that, under the selected conditions of testing, the 310 stainless steel was not protected by hot-dip aluminum coating. Prior heat-treatment of the coatings had no considerable effect. Confirmation that these results are indicative of what may be expected in service is provided by the example of a boiler service test. Other burner tests and tentative results from shipboard service trials also are consistent with these findings.

In spite of this excellent agreement, it is considered that the test methods used were over-simplified and did not cover adequately the whole range of variables possible in service exposure. In the present investigation such a procedure was justified by expediency but there was the safeguard of comparing the results with others obtained by different procedures. For a thorough, extensive investigation of resistance of materials to fuel-ash attack, a carefully designed burner test rig in which specimens would be exposed to conditions closely simulating those prevailing in service must be regarded as a necessity. Furthermore, an adequate program to correlate the results obtained in such a rig with results of actual service trials would have to be undertaken before the former could be accepted with confidence.

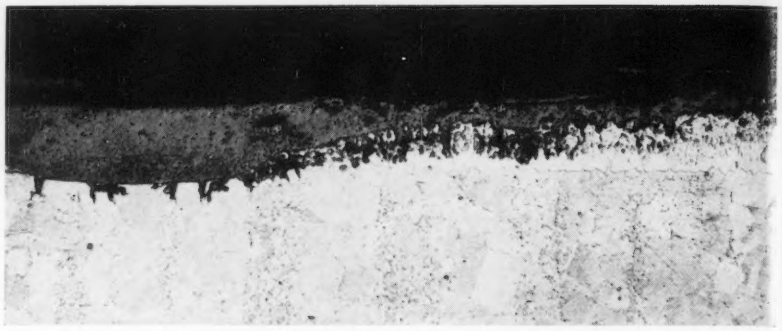

Figure 6—Penetration of coating in eight days at 1700 F (see text and Figures 1 and 2). Original magnification 100X; actual magnification after reduction for engraving purposes, 72X. Etched.

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Any discussions of this article not published above will appear in the June, 1958 issue

Cavity Formation in Iron Oxide*

By D. W. JUENKER,[1] R. A. MEUSSNER[2] and C. E. BIRCHENALL[3]

Abstract

A study is made of the deep oxidation of iron at high temperatures and in an oxygen atmosphere. Large cavities are always found in specimens whose total oxygen content has been brought to that of FeO. The scale thickness of such specimens, along with other evidence, implies that FeO is plastic in the temperature range in which it is stable, while one or both of the higher oxides is relatively rigid. Evidence is presented which indicates that iron is transported to its surrounding scale with little or no body diffusion path; the mechanism involved is not identified. An expression, analogous to the parabolic rate law for scaling of plane specimens, is developed for application to cylindrical geometry. 3.2.3

Introduction

AT TEMPERATURES above approximately 600 C, iron is capable of forming three stable oxides: wüstite (FeO), magnetite (Fe_3O_4), and hematite (Fe_2O_3). Wüstite, which has a stoichiometric deficit of iron throughout its broad composition range and is unstable at low temperatures, ordinarily is the major constituent of the high-temperature oxide scale. Detailed investigations of the growth mechanisms of the three phases and of the transport characteristics of their components have been carried out by Davies, Simnad, and Birchenall,[1] and by Himmel, Mehl, and Birchenall.[2] As a result of these studies it appears that practically all of the material transport responsible for the growth of wüstite, and probably of magnetite as well, is the self-diffusion of iron in the oxide lattice.

Some research activities by the present authors have been directed toward an improvement in the accuracy of determination of the rate of growth of magnetite on the lower oxide. In the course of this program, attempts were made to grow suitable wüstite specimens from pure iron by direct oxidation in an oxygen atmosphere. The occurrence of an unexpected set of phenomena at this stage of the experiment has led to the study which is the subject of the present paper.

In general, the aim of this work is a qualitative investigation of the oxidation of iron in pure oxygen at temperatures between 700 and 1000 C. Under these conditions, all three oxide phases normally are present. The progressive diminution of the iron core due to the outward migration through the oxide lattice results in stresses throughout the sample. Unless the oxide scale is sufficiently plastic to follow the recession of its iron foundation, these stresses must be relieved by internal fracture of the oxide or by separation, at least locally, of the scale from the iron. The continued oxidation following the latter type of separation is the particular object of the present study; in some respects the resultant findings appear to be related to those achieved by Pfeil[3] and by Dunnington, Beck and Fontana[4] under similar circumstances. Other aspects of the problem which have not been reported hitherto are also of general interest.

Experimental Method

Since the deep scaling of iron at high temperature and in the presence of an oxygen-rich atmosphere involves a number of competing processes, two specimens subjected to conditions as closely identical as is practically feasible in general will not exhibit quantitatively identical behavior. Furthermore, it is obvious that a cross-sectional examination of a sample at some stage in the oxidation process precludes the continuation of the process on the same sample. Hence the present experiment consists necessarily in a summary of observations made visually and micrographically on many individual specimens, conclusions being drawn for the most part from consistent qualitative similarities. The sole quantity which has been measured as a function of time during the oxidation itself is the specimen's weight, changes in which can be correlated with physical characteristics of the scale.

The principal component of the oxidation apparatus used in the present work resembles that employed by Davies, Simnad, and Birchenall.[2] It consists of a 50-mm fused silica reaction chamber about 18 inches in length which is closed at its lower end. At its other end it is connected by means of a ground standard taper joint to a four-foot length of Pyrex cylinder. The latter is capped with a short Pyrex section into which is introduced a windlass arrangement, also by means of a ground joint. A nylon thread wound on the windlass supports a sensitive spring of low temperature coefficients of expansion and elasticity. The sample to be oxidized is suspended from this spring by means of a platinum filament.

The silica-end of the assembly is enclosed in a vertical Kanthal-wound tubular furnace. Interior and exterior radiation shields and a water cooling jacket of copper tubing are situated at the top of the furnace to minimize temperature variations and convection currents within the oxidation chamber and spring enclosure. Measurements taken from a thermocouple attached to a "dummy" sample showed that, in the region within 8 cm from the bottom of the reaction chamber, the temperature varied by less than two degrees C in a thousand, in both distance and time.

The springs used to follow the change in weight of the specimens during oxidation were age-hardened one-inch helices of 10-mil Ni-Span-C wire. These were normally of about 50 turns, with a sensitivity of about 5 mg-mm^{-1}, but for heavier samples 25-turn springs were used successfully, despite the consequent halving of the sensitivity. By cathetometer measurements of the spring length, weight changes could be followed to within about 0.05 percent of the total load. Five-mil platinum wire was used to suspend a specimen from the spring, since lighter filaments showed a tendency to part under loaded conditions at high temperatures.

Vacuum and gas connections to the reaction chamber were such that the chamber could be evacuated, flushed with inert gas, or loaded with the desired oxidizing or reducing atmosphere whenever such operations were required. In the present work, only oxygen and argon were used, and these were subjected to elementary purification by passage through towers of Ascarite and Drierite. Three oxidation units were included in the complete apparatus, so that several samples could be studied simultaneously under different conditions.

The iron samples studied were of two types: flat specimens, 1 inch x ¼ inch x 1/16 inch, of Westinghouse Puron; and round Armco Iron rod of various diameters, 1 inch to 1½ inches long. Before use, these were ground, washed in acetone, and cleaned for three hours in wet hydrogen and three hours in dry hydrogen at about 850 C. Subsequently, they were not permitted to contact possible sources of organic contamination. Each sample was weighed initially, attached to a spring suspension, and hung in the cold zone of a reaction chamber. With the furnace at the desired temperature, the chamber was evacuated and flushed several times with argon. The sample was then lowered into the uniform temperature zone of the furnace and permitted to come to temperature in the argon atmosphere. During this period the initial load was determined by measuring the spring extension. After some ten minutes had been allowed for temperature equilibration, the reaction chamber was evacuated and oxygen admitted. Throughout the oxidation period, pressure was maintained at one atmosphere by causing the oxygen to flow slowly through a butyl phthalate bubbler. The weight gain of the sample was monitored until the desired oxygen content had been achieved. At this time the sample either was removed to the cool upper portion of the reaction vessel, or was left in the hot zone for homogenization, with the oxygen being replaced by argon. During such homogenization heat treatments, no weight changes were detected in the specimens studied.

For photomicrographic observation, samples were mounted in an epoxy resin (Epon 828), ground on abrasive paper, and fine-polished on a cloth wheel using magnesium oxide, alumina, or chrome oxide abrasives. A saturated solution of stannous chloride in ethyl alcohol or a 4 percent solution of picric acid in ethanol was used for etching purposes.

★ Submitted for publication November 9, 1956.

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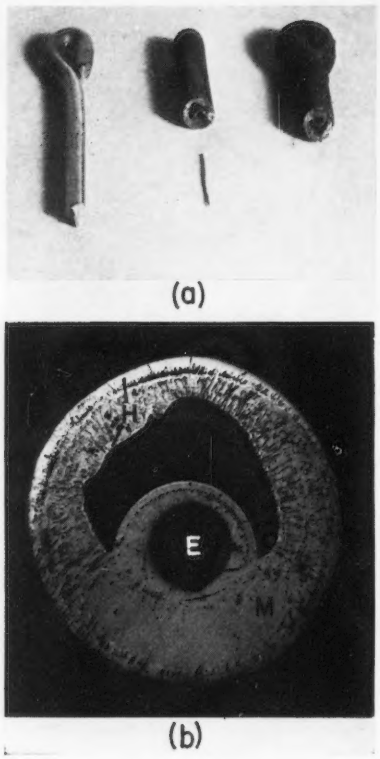

Figure 1—Locally adherent scaling in a cylindrical sample oxidized to 27.2 percent oxygen by weight at 850 C.
(a) Oxide broken open, with lower section (center) of the residual filament removed. A 1/16 inch diameter (D_o) Armco Iron hook, similar to that from which the sample was grown, is shown for comparison.
(b) Photomicrograph of a cross-section of the same sample mounted without the central filament in Epon (E). The lightest material in the scale is hematite (H), the remainder principally magnetite (M). Stannous chloride etch. Original magnification, 24X; actual magnification after reduction for engraving purposes, 20X.

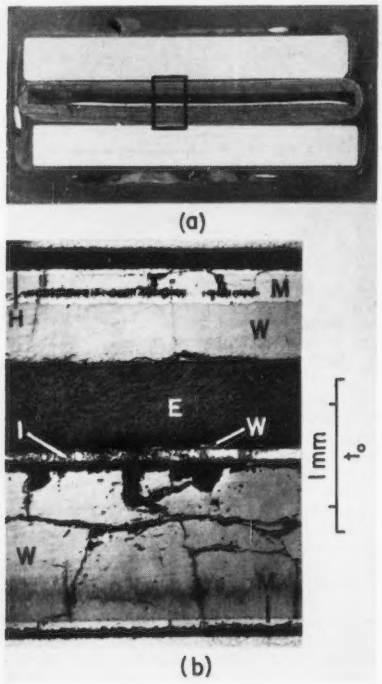

Figure 2—Locally adherent scaling in a flat specimen oxidized to 23.9 percent oxygen by weight at 900 C. The original blank was Puron, of thickness (t_o) 1/16."
(a) Sample mounted edgewise between blocks of iron and ground to reveal longitudinal cross-section.
(b) Photomicrograph of the area outlined in (a). A layer of hematite (H) covers the exterior of the sample, being most noticeable on the uppermost edge; a small amount of wustite (W) is observable adhering to the upper side of the central residual iron strip (I). M and E designate magnetite and the Epon mount. Stannous chloride etch. Original magnification, 20X.

Results

The phenomena observed in the present work can be assigned arbitrarily to three categories, or to combinations of the three:

(a) Incomplete separation of the scale from its iron base, with oxidation proceeding at the remaining regions of adhesion. This locally adherent oxide has been treated at length by Dunnington, Beck and Fontana.[4]

(b) Complete separation of iron and scale, followed by continuing iron and oxygen transport as evidenced by the progressive depletion of the free iron supply. In his early work on high-temperature oxidation of iron and steel, Pfeil[3] has described a "loose core" scale which is undoubtedly of this type. Although Pfeil's evidence for this behavior was admittedly fragmentary, it now appears to be well founded. On the other hand, his generalization that the oxide grows in three distinct laminae (related to, but not to be identified with, the three oxide phases) does not seem valid.

(c) The peeling off of a thin layer of adherent scale as the iron separates from the oxide. This consists in a localized rupture within the scale layer near, and parallel to, the metal surface, leaving on the iron an adherent coating which joins elsewhere to the main oxide body.

What could be considered a fourth category is the deep rupture of the oxide, permitting gaseous oxygen to enter the sample's interior uninhibited. Considered by itself, however, this is irrelevant to the present study and will not be discussed.

In the photomicrographs to be shown subsequently, the various oxide phases generally can be identified by the shades of grey brought out in etching: these range from light hematite (H), through intermediate magnetite (M), to dark wüstite (W). The mounting resin (E) ordinarily is darkest of all and can be recognized by its mottled appearance. Free iron (I) is seen in the lightest portions of the photographs. Proeutectoid magnetite is visible in some areas where it was precipitated from oxygen-rich wüstite during cooling. The oxygen contents in weight percent for the three oxides are: wüstite, 23.5 to 24.4; magnetite, 27.7; and hematite, 30.1.

Locally Adherent Oxide

Cylindrical Sample. Figure 1 shows an example of the locally adherent scale in a cylindrical sample. Two stages of the oxidation process are immediately evident. First, the iron separated from the oxide over all but a small segment of its circumference and local oxidation progressed. Secondly, free oxygen was admitted to the resultant inner void, probably by way of a radial fissure, and oxidation proceeded rapidly within the sample. This is verified by the light hematite border surrounding the crescent-shaped void in the photomicrograph in Figure 1. Except for the outer layer of hematite, the remainder of the oxide is almost completely in the magnetite phase, the sample having been oxidized to 27.2 percent oxygen by weight.

The process by which the remaining free iron was depleted is not so readily apparent. It is probable that a second separation took place, with local adhesion accounting for the further oxidation. This need not have been the only mechanism in operation; as shown in the macrophotograph in Figure 1, only a fine residual oxidized filament remained of the original iron core. This filament was separate from the body of the scale; some light connection might have been broken in opening the sample for inspection, but had such a connection existed, it must have been too tenuous to support considerable material transport. The filament was removed before the sample was mounted for photomicrography.

Flat Specimen. Another sample of the adherent oxide is the flat specimen in Figure 2, which was oxidized to 23.9 percent oxygen by weight. The separation of oxide and iron was complete along one face of the sample, with oxidation proceeding along the other. The separated scale contains thick layers of higher oxides, while the adherent oxide is almost entirely wüstite, adjoining a thin slab of iron which bears fragments of adherent wüstite on its free side. It is to be noted that the internal void is considerably narrower than the original sample thickness. The uniformity of the oxygen concentration gradient in the wüstite, as shown by the magnetite precipitation, is sufficient evidence that the many cracks in the thick side of the scale occurred in cooling, sectioning, or mounting, and not in the oxidation process.

Oxygen Content Versus Time. Figures 3 through 10 show examples of cylindrical specimens for which detailed data were taken for oxygen content versus time at 950 C. Under the simplifying assumptions that the scale is pure wüstite coated with a negligibly thin layer of the higher oxides, that the scale is sufficiently plastic to follow the recession of the iron substrate, and that porosity and end effects can be neglected, it is possible to plot weight change as a function of time for cylindrical samples on a universal graph. The mass change ΔM, when normalized to unit initial radius R_o and surface area A_o, is a monotonically increasing function of the quantity $(K_p/R_o)t^{1/2}$, where t is the time and K_p is the parabolic rate constant determined experimentally for plane specimens. The derivation of this relationship is given in the appendix. The scale separation which has been shown to occur renders improbable the realization of an experimental plot which will follow the theoretical prediction for any considerable length of time. However, the plotting of $\Delta M/(R_oA_o)$ versus $(K_p/R_o)t^{1/2}$ retains its usefulness as a means of comparison between cylindrical specimens of different diameters, oxidized at different temperatures. The values of the parabolic rate constant employed here were interpolated from the data given by Davies, Simnad, and Birchenall[2]; present results are in agreement with these data, as is

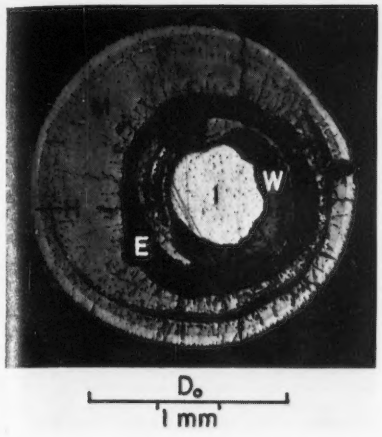

Figure 3—Cross-section of a cylindrical Armco Iron specimen oxidized to 24.2 percent oxygen by weight at 950 C; the original diameter D_o was ⅛ inch. The light material on the periphery and bordering the voids is hematite (H). The central core is free iron (I) with adherent wustite (W) scale on the right and magnetite (M) on the left. The remainder of the scale is magnetite. Picral etch. Original magnification, 12X.

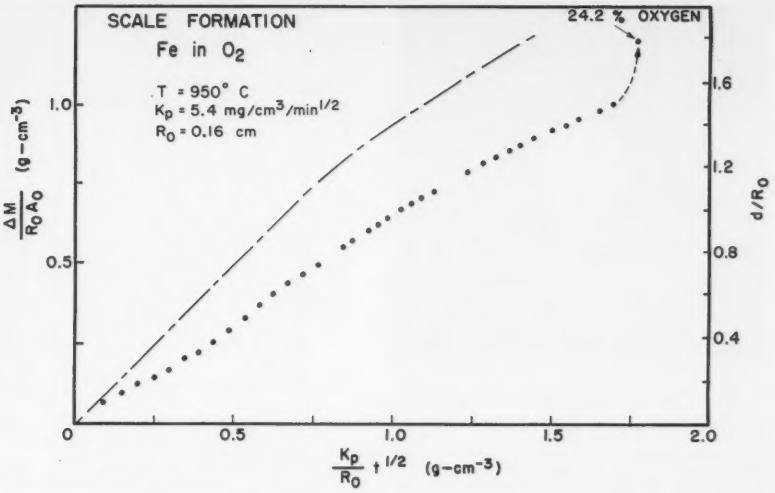

Figure 4—Progress of the oxidation of the sample in Figure 3. The change in mass $\triangle M$, normalized to unit initial radius R_o and surface area A_o is plotted against $(K_p/R_o)\ t^{1/2}$, where K_p is the known value of the parabolic rate constant and t the time. The broken line represents the theoretical behavior for an ideally plastic, adherent, and non-porous scale on a cylinder of infinite length. The ordinate on the right gives the thickness d of such an ideal scale.

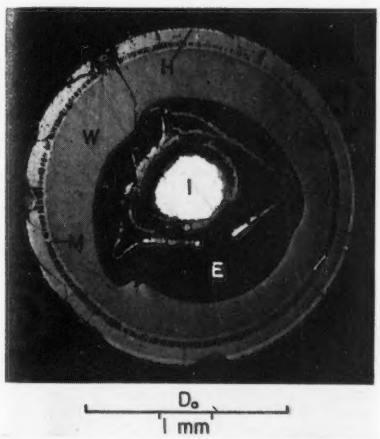

Figure 5—Cross-section of cylindrical Armco Iron specimen oxidized to 23.9 percent oxygen by weight at 950 C; the original diameter D_o was 5/32 inch. light outer border is hematite. The wustite-magnetite magnetite boundary lies just within the porosity ring, and the central light region is free iron (I). Picral etch. Original magnification, 12X.

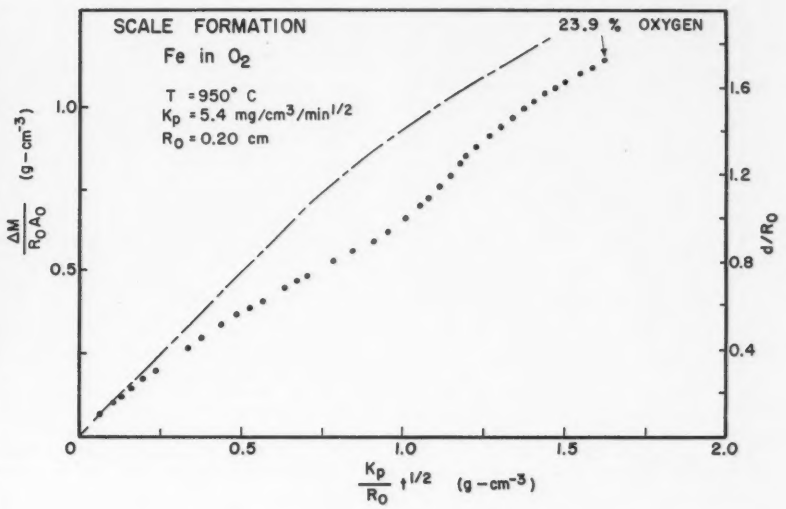

Figure 6—Progress of the oxidation of the sample in Figure 5.

shown by the approach of the slopes of experimental curves to those of the theoretical lines near zero time in Figures 4, 6, 8 and 10.

Tests on ⅛-Inch Diameter Sample. The experimental points shown in Figure 4 are for a ⅛-inch diameter sample. They fall away from the theoretical expectation given by the dashed curve almost immediately after the beginning of oxidation, revealing the formation of porosity or a detachment of the scale. The acceleration of the oxidation rate beginning at an abscissal value near 0.4 gm-cm^{-3} is indicative either of a breakthrough of gaseous oxygen to the sample's interior or of a healing of the porosity or separation. The subsequent deceleration period can be attributed to further porosity formation or detachment, and the final rapid increase in mass almost certainly is due to an oxygen leak.

Specific connection of these developments with the features of the photomicrograph in Figure 3 is not obvious at first, since the outward-growing nature of the scale makes it difficult to determine the chronological order in which the visible characteristics were formed. However, the increase in mass at an abscissa of 1.7 gm-cm^{-3} corresponds roughly to the oxygen content of the scale immediately surrounding the residual iron core. Furthermore, the outermost, semi-circular, void is bordered with hematite, and a thin hematite ring is visible, partially completing the circle of which the void is an arc.

These facts imply that the outer void was formed first as in the case of the specimen in Figure 1, thus accounting for the initial deceleration of the oxidation; a subsequent oxygen leak continued the oxidation and probably at the same time healed the porosity at the position of the partial hematite ring previously mentioned—hence the early acceleration period. Thereafter a second detachment occurred, with little or no contact left between iron and scale, since there was not sufficient iron transport to prevent the complete conversion of the wüstite outer shell to magnetite; this process was evidently going on during the long second deceleration period represented in the plot of Figure 4. Finally, the whole process seems to have been repeated on the remaining iron core when a second oxygen leak promoted the last acceleration.

Tests on 5/32-Inch Diameter Sample. The sample described in Figures 5 and 6 was originally 5/32 inch in diameter.

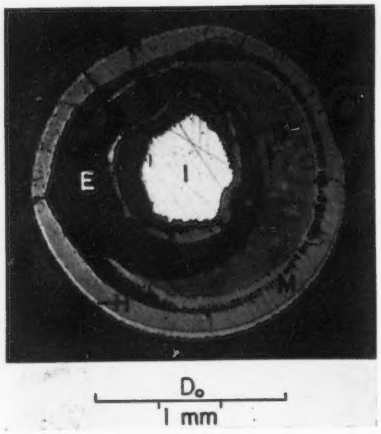

Figure 7—Cross-section of a cylindrical Armco Iron specimen oxidized to 21.9 percent oxygen by weight at 950 C; the original diameter D_o was ⅛ inch. The light outer border is hematite. The wustite-magnetite boundary lies just within the porosity ring. Visible in the wustite field is some magnetite, precipitated during the slow cool. The light central region is free iron. Picral etch. Original magnification, 12X.

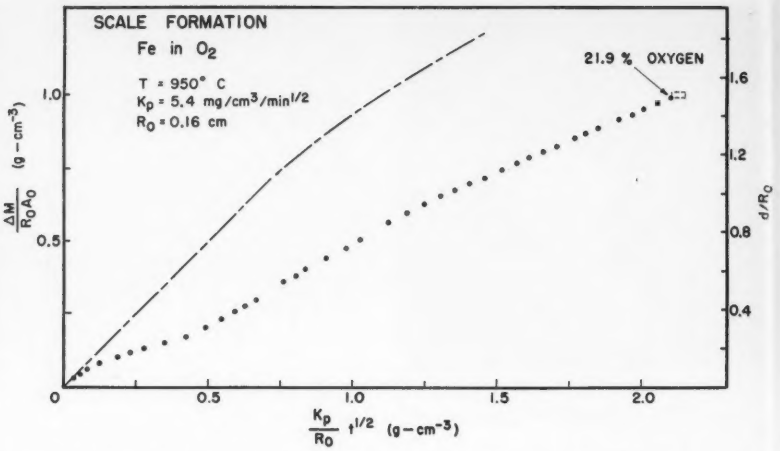

Figure 8—Progress of the oxidation of the sample in Figure 7. The rectangle at the upper end of the dotted data line indicates the uncertainty in the final measurement.

Here there are no indications of an early scale separation. The first deceleration period doubtless is due to the formation of the outer porosity ring, followed eventually by a large-scale withdrawal of the iron from its scale envelope. The slow acceleration near 1.1 gm-cm^{-3} is not attributable to an oxygen leak to the interior, since the wüstite surrounding the central void is solid and pore-free, and has no traces of higher oxides on its inner border (a uniform distribution of precipitated magnetite is visible throughout the wüstite area at higher magnifications); more likely it corresponds to a partial healing of the outer porosity ring.

An interesting feature in Figure 5 is the wüstite "rind" surrounding the remnant iron. Judging from other evidence, as will be shown in Figure 11b, this sheath probably is continuous in the axial direction. It has one connection with the body of the wüstite scale; multiple connections have not been observed in this or any similar specimen. The over-all appearance suggests that the iron-to-oxide bond was sufficiently strong that a thin layer of wüstite was peeled off when the iron was forced by internal stresses to separate from the scale. A final observation to be made in connection with Figure 5 is that a large amount of iron appears to have been transported to the oxide without an appreciable diffusion path.

A similar situation is seen in Figures 7 and 8, with a sample originally ⅛ inch in diameter. The outer porosity ring is more extreme here, amounting to complete separation around one side of the sample; this seems to be characteristic of smaller specimens. The incidence of large concentrations of magnetite in the wüstite region is due to an unintentionally slow cooling.

Completely Separated Scale

The oxidation of a 3/16-inch iron cylinder is described in Figures 9 and 10. Both photomicrograph and mass versus time plot disclose a behavior nearly identical with that shown in Figures 3 and 4, up to the final step. Here, after the iron residue has been oxidized to some extent by local adhesion, it has broken away completely for the last time, but has continued to be transported to the oxide. That this is so is borne out by the fact that while the topography of the iron core appears to fit that of the adjacent wüstite, there is a 20 percent dimensional difference which cannot be explained by any reasonable degree of contraction in cooling. A final observation can be made with regard to Figure 9; unlike the samples shown in Figures 1, 3, 5, and 7, the inner diameter of the outer oxide shell is 20 percent smaller than the original diameter of the iron blank.

Further examples of the completely separated, "loose core" type oxide are shown in Figures 11 and 12. The specimen in Figure 11a was oxidized at 930 C to a 16.2 percent oxygen content. Removal of the one end revealed a cylindrical oxide shell of uniform wall thickness and internal diameter slightly smaller than the original diameter of the iron blank (⅛ inch), a duplicate of which is shown for comparison. The internal geometry of the shell was found to be identical with the external features of the original iron, even to a small solid cylinder of oxide in the position formerly occupied by the suspension hole in the iron blank. Within the shell was the lightly oxidized iron residue, completely detached and still cylindrical in form, but bearing on its surface a slight ridge of oxide running parallel to its axis. This was probably the last line of the oxide-iron contact. The ridge is barely visible in the photograph.

Another uniform detached scale is shown in Figure 11b. In this case, the original iron diameter was 1/16 inch and the oxidation was carried out at 850 C to a 25.5 percent oxygen content, followed by a homogenization treatment in argon at the same temperature for a time approximately equal to that occupied by the oxidation process (33 hours). The grainy surface appearance of the oxide is a consequence of the homogenization. Again the uniform cylinder wall has resulted and the fine residual central filament of lightly oxidized iron is encased in a fragile sheath of irregular cross-section, reminiscent of the thin oxide shells described in connection with Figures 5 and 7. Central filament, sheath, and outer shell are mutually separate.

A behavior of some interest is typified by the samples shown in Figure 12. Together with a comparison iron blank are two flat specimens with their longer edges ground away to reveal their interiors. Both are 24.7 percent oxygen by weight, one having been oxidized at 800 C and homogenized for 70 hours in argon at the same temperature, the other oxidized with the temperature varying between 700 and 925 C and homogenized for 50 hours, also at 800 C. Aside from a slight irregularity at one end of the latter specimen, the geometrical similarity between the iron blank and the internal cavities in these specimens is remarkable. Oxide tubes replace the holes drilled for suspension purposes; corners remain sharp and wall surfaces very nearly flat (the broad surfaces perpendicular to the plane of the photograph are bowed slightly inward, the transverse widths of the cavities being up to 10 percent less at the centers than at the ends). Moreover, the walls are similar in thickness on either side of the cavity, which would seem to eliminate the possibility that the scale grew by adhesion of the iron to one side or the other. It should be added that there was no perceptible loss of internal material during the grinding process.

Inert Marker Studies

Some information regarding the transport of iron to the detached scale can be obtained from the study of the movement of inert markers. Examples of such a study are shown in Figures 13 and 14. In the partially oxidized flat specimen in Figure 13, scale separation was initiated at the upper right and lower left corners, so that detached and adherent oxides appear in neighboring regions on the same side of the sample. Four transverse stripes of chromic oxide were painted on each face of the iron blank before oxidation was begun. Since these should remain fixed in the oxide lattice, and diffusion of iron is the principal transport mechanism involved, one expects to find these markers close to the oxide-iron interface at any stage of the scale growth. This is seen to be the case in the photomicrograph of the adherent portion of the scale (Figure 13b), where the inert marker appears at the interface.

In the detached scale, on the other hand, the markers are separated from the void between the iron and the oxide

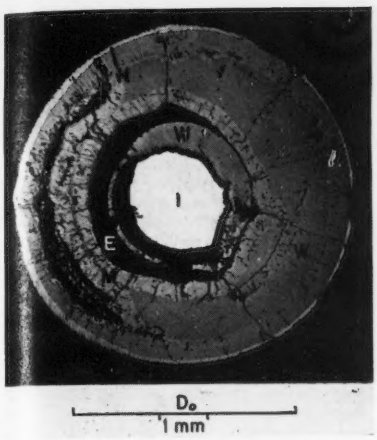

Figure 9—Cross-section of a cylindrical Armco Iron specimen oxidized to 23.9 percent oxygen by weight at 950 C; the original diameter D_o was 3/16 inch. Hematite borders the outer surface, the porous region on the left, and the inner surface of the larger oxide shell. The remainder of this shell is magnetite, except for a reversed "C"-shaped wustite region on the right. The smaller interior shell is principally wustite, surrounding a separated free iron core. Picral etch. Original magnification, 12X.

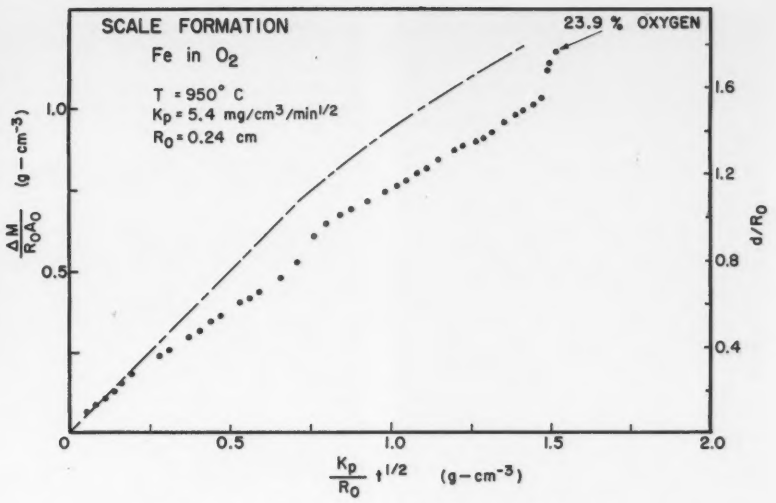

Figure 10—Progress of the oxidation of the sample in Figure 9.

by a considerable thickness of wüstite shown in Figures 13c and 13d. The buried markers are generally easy to locate, and are always within the detached portion of the scale. Markers in the adherent region are seldom found, probably because their position at the interface permits them to disintegrate when the sample is cooled and cracking occurs. Oxygen comprises 13.8 percent of the total weight of the sample in Figure 13, which was oxidized at 900 C.

Another marker-bearing sample is shown in Figure 14. In this case, oxidation was carried out on a flat Puron blank at 900 C to an over-all wüstite (24.0 percent oxygen) composition, and the sample was homogenized at the same temperature for 62 hours. The cross-sectional plane pictured in Figure 14a discloses a cavity much smaller in transverse dimension than those in Figures 2 and 12. Two markers were located in the oxide area lowermost in Figure 14a. Both were imbedded within the wüstite, as shown in Figure 14b. Of four markers painted on the upper side (relative to the photograph), only one was found, but not positively identified. It lay close to the edge of the central cavity, as shown in Figure 14c.

Discussion

Plastic Properties of the Iron Oxides

The formation of large voids within the high temperature scales grown on iron at high oxygen pressures appears to be inevitable if the scale remains sound. In the case where oxidation proceeds by means of local adhesion between iron and scale, the voids can be comparable in volume to the original iron sample. The explanation lies in the iron diffusion process principally responsible for the oxide growth; this already has been recognized by Dunnington, Beck, and Fontana.[4] Implicit in the phenomenon, however, are the plastic properties of the scale at elevated temperature. The scale is sufficiently plastic to follow the recession of the free iron surface until a considerable oxide wall thickness has been built up. The plasticity, however, is not great enough to relieve the stresses which eventually cause scale separation.

Specimens of wüstite have been grown, solid and uniform throughout save for minute V-shaped voids near their centers.[5] However, these were formed by oxidation in an H_2-H_2O atmosphere of proportions which provided a partial pressure of oxygen in equilibrium with that of wüstite.[(9)] Under these conditions, no higher oxides could be formed. It is probable, therefore, that while wüstite is sufficiently plastic to follow the inward movement of the oxide-iron interface, it is held back by the coating of one or both of the higher oxides which form at high oxygen pressures. Further, one can assign a coincidence between the occurrence of scale separation and the time at which the thickness of higher oxides, coupled with the surface geometry, is sufficient to inhibit the wüstite flow.

The relative rigidity of the higher oxides with respect to wüstite also may have some bearing on the formation of orderly porosity in the magnetite regions of the scale. Such porosity rarely occurs along the plane sides of a flat specimen, where no great additional strain is imposed on the superposed oxide as a result of the inward motion of the wüstite-iron interface. But a ring of porosity is always found at the corners of a flat scale specimen[6] and in samples of circular cross-section, to a degree which lessens as the surface curvature decreases and approaches that of a plane. Note in this connection that in samples of larger diameter (for example, those of Figures 6 and 10) oxidation follows the theoretical expectation for comparatively longer times. In cylindrical samples, the porosity is annular, and is situated close to the magnetite-wüstite boundary. A rigorous analysis of this situation has not been undertaken, but it may be possible to relate the porosity formation to the radius of curvature of that boundary.

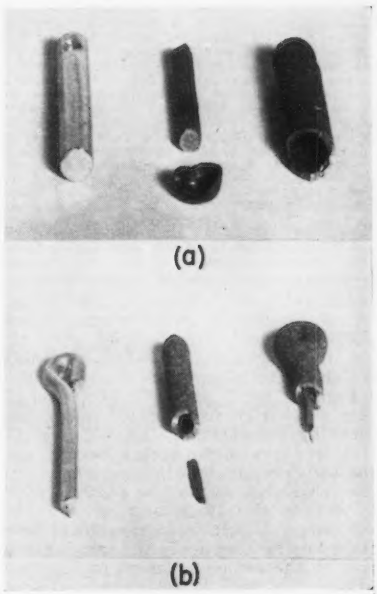

Figure 11—Uniform, completely detached scale on cylindrical Armco Iron specimens, with duplicates of the original blanks shown for comparison.
(a) One-eighth inch sample oxidized to 16.2 percent oxygen by weight at 930 C. The lower end (lower center) has been cracked off to show its interior, and the core (upper center) withdrawn from the shell (right).
(b) One-sixteenth inch sample oxidized to 25.5 percent oxygen by weight at 850 C. The sample has been broken open and the lower section (lower center) of the oxide "sheath" covering the residual filament (lower right) has been removed. The grainy appearance of the oxide surface results from a 33-hour homogenization treatment in argon at 930 C.

Several other results emerge from the consideration of a plastic wüstite within a rigid magnetite "cage." Oxide-iron separation will occur early in oxidation at the corners of a rectangular specimen where stresses become extreme; this will leave the geometry of such corners in the final oxide cavity very nearly the same as existed in the original iron blank. On the plane surfaces, meanwhile, the oxide will follow the recession

(9) The formation of sound wüstite under these conditions has been confirmed by J. D. Mackenzie in the Forrestal Research Center laboratories of Princeton University. The results of this study were published on pages 783t-785t of the December, 1957 issue of *Corrosion*.

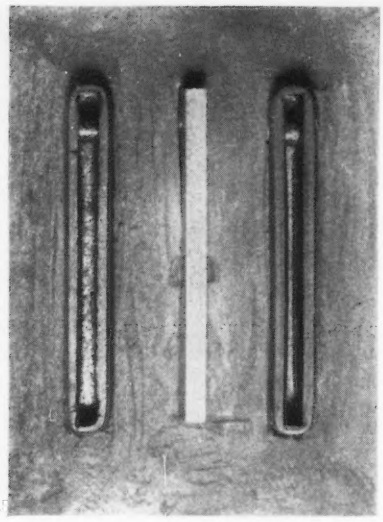

Figure 12—Uniform oxides grown from flat Puron blanks. A duplicate of the original blanks, 1/16 inch thick and 1 inch long, is shown edge-on (center). A longer edge of each of the samples has been ground away to reveal the interior cavities; both samples are 24.7 percent oxygen by weight. That on the right was oxidized, then homogenized in argon for 70 hours, both at 800 C; the sample on the left was oxidized at temperatures varying between 700 C and 925 C and homogenized for 50 hours at 800 C. The solid cylinders of oxide near the tops of the samples' interior cavities occupy the previous positions of holes drilled in the blanks for suspension purposes.

of the iron until the suspension-type stresses originating in the rigid magnetite-lined corners cause the corner separation to propagate along the plane surfaces. Cavity surfaces, therefore, should be bowed inward to some extent. Both of these characteristics are found in all flat specimens in which separation is complete along one or more faces (Figures 2, 12, and 14). Moreover, in samples which have been homogenized at high temperature (e.g., Figure 14) the tendency of the scale's internal void to collapse can be attributed to plastic flow, which also may be partly responsible for the small size of the void shown in Figure 9. It would not seem fair, however, to apply the same reasoning to the behavior shown in Figure 2, where the void is somewhat narrower than the original iron blank.

Finally, the plasticity hypothesis helps in supplying a tentative explanation of the wüstite sheath enveloping the residual iron core, as shown in Figures 5, 7, and 11. As previously suggested in the discussion of those specimens, separation could take place within the wüstite layer, given a strong adherence between wüstite and iron. Under this condition the wüstite would need to be rather highly plastic to submit to the subsequent deformation without experiencing disruptive stresses. There is no evident reason for the intra-wüstite separation to be anything but complicated, hence the observation that the oxide sheaths shown in Figures 5 and 7 have single connections with the body of the scale need not be general.

Transport Mechanisms in Separated Scales

Pfeil[8] has described experiments in which he observed material transport between oxides and iron, and between oxides of different oxygen contents, out of contact and in inert atmospheres and in vacuum. The samples were studied in the temperature range of the present investigation. These studies indicated a net current of oxygen toward the more iron-rich sample, apparently due to the higher vapor pressures of oxygen of the higher oxides.

In the present work, an explanation is sought for the reverse process which takes place in the continued growth of a completely separated scale. The evidence in Figures 5, 9, 11, and 12 is in favor of a net iron current from the separated iron core, even though the latter may be partially oxidized. Four remote possibilities for such a transport suggest themselves: (1) Iron evaporation, (2) Volume diffusion through small localized connections remaining after the otherwise complete scale separation, (3) Diffusion of iron over the inner oxide surface, originating at the points of physical contact between iron and scale, and (4) Oxide vapor exchange.

The first of these processes can be ruled out almost immediately on the basis of the time involved. For the samples shown in Figure 12, with the liberal assumption that half of the iron mass had been dissipated before separation occurred, an additional time of the order of thousands of hours would have been required to evaporate the remainder. An order-of-magnitude check has been made on the known value of the evaporation rate of iron[7] by the following method. A Svea metal (Swedish iron) wire, 0.0145 inch in diameter, was cleaned, hydrogen-fired, and mounted in a Pyrex tube which was evacuated below 10^{-7} mm of Hg. The wire was heated to a temperature between 900 and 1000 C by the passage of direct current. The temperature was held constant by maintaining at a constant value the product of the heating current by the cube of the voltage drop between two potential leads attached to the wire, a few centimeters apart. The change in wire radius due to evaporation was then followed by measurements of the resistance between the two potential leads, and converted to evaporation rate. The latter was found to be approximately 10^{-8} gm-cm^{-2}-sec^{-1}, which is of the expected order of magnitude.

Iron is transferred continuously to the scale through areas of local adhesion. Such transfer continues after the oxygen atmosphere has been removed, as witnessed by the homogenized specimens in which residual iron was found to be completely absent. However, it does not seem that such a process would result in an oxide shell of uniform wall thickness. The sample shown in Figure 2, for example, contains nearly twice as much oxygen on one of its sides as on the other. Unless the oxygen itself can migrate, the walls would be of unequal thicknesses after a homogenization treatment.

Small bridges may exist between scale and separated iron, as shown in Figures 5 and 7, but their cross-section and extreme length would render negligible the material diffused through them within the total time of oxidation. It is probable that the surface diffusion hypothesis must be ruled out also. Granting even that iron can migrate swiftly over an oxide surface (the rate is unknown), the contact between iron and oxide necessary to initiate a large-scale migration of this type would need to be so extensive that it would amount to adhesion. In this case, as well as in that of the aforementioned bridge connection, the strong local oxidation would result in a non-uniform scale thickness, which has not been observed.

Aside from the objections mentioned, all three of the foregoing mechanisms share a common failing in that they do not explain the positions of inert markers in detached scale. Free iron on the inner oxide surface, regardless of the manner of its deposition, should diffuse outward past the markers. The presence of the latter buried within the scale indicates that oxide, or gaseous oxygen, was deposited over them. The effect of the presence of gaseous oxygen has been shown in the case of cylindrical samples: a new shell of oxide rapidly forms on the free iron surface. This is not observed, for example, in the sample in Figure 13.

It has been reported by Pfeil[8] that iron heated in vacuum in the neighborhood of oxygen-rich wüstite will acquire a wüstite scale, with no net loss of iron. The vacuum conditions for Pfeil's investigation were not precisely described, but could not have been favorably comparable with those existing in a void within a sound oxide. Aside from this difficulty, Pfeil's results could be interpreted on the basis either of the evaporation of oxygen from the oxide, or of a molecular vapor exchange of a higher oxide for a lower, balanced with respect to iron. In the latter regard, there is perhaps some small possibility that one of the iron oxides in molecular or complex form is sufficiently volatile that the addition of oxygen or a higher oxide in molecular amounts to a clean iron surface will result in a net release of iron from the free surface. Such a reaction is highly dubious, and while it would account for the buried markers in the present study, the large sizes of the voids in many of the samples would remain unexplained.

The question of the reaction-sustaining material transport has been brought up recently by Pfeiffer and Ilschner.[9] Although they interpret their results differently, the two examples they show of oxidized iron wire bear strong resemblance to some of the specimens illustrated in the present work: one a uniform, completely detached scale, and the other a locally adherent oxide. While the latter sample was oxidized near 600 C, close to the borderline for wüstite formation, present evidence would indicate that physically similar scale growth can occur at higher temperatures, and hence, that the difference in appearance of the two specimens shown by Pfeiffer and Ilschner is fortuitous rather than temperature-determined as they suggest. The same authors state that they could not observe the expected decrease in growth rate due to porosity formation (or scale separation). As can be seen in the mass versus time data shown here, such a deceleration can be difficult to distinguish from normal growth in a cylindrical sample unless extremely detailed data are taken.

Summary

Attributing plasticity to FeO at high temperatures accounts for the majority of the features encountered in the deep scaling of iron in an oxygen atmosphere. However, the consumption of the remnant iron in loose-core specimens lacks interpretation. The following investiga-

tions would be helpful in settling both matters:

(a) **Determination of the plastic properties of the oxides of iron at high temperatures.** In line with such a determination, it would be worthwhile to obtain mass versus time data for the oxidation of long cylinders in an atmosphere equilibrated for oxygen-rich wüstite. Under these conditions, and provided that the oxide is indeed plastic, the reaction would approach the ideal conditions for which the theoretical expression given in the appendix applies, and the complications introduced by edge effects in rectangular specimens would be avoided.

(b) **Measurement of the surface diffusion rate of iron on an oxide surface, and further experiments of the type employed by Pfeil on material transport between oxides.** Such measurements would be facilitated by radioactive tracer techniques.

(c) **Accumulation of data on inert marker movement in flat specimens oxidized in an oxygen atmosphere.** In this case, the flat geometry is preferable to the cylindrical on the grounds of less ambiguity in the interpretation of results.

Finally, it goes without saying that any information gained by tracer studies, or with such methods as field emission microscopy, on the chemistry of oxygen on a clean iron surface would be invaluable.

Acknowledgment

This research was supported by the United States Air Force, through the Office of Scientific Research of the Air Research and Development Command.

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APPENDIX—Parabolic Growth Problem in Cylindrical Geometry

The assumptions necessary to the formulation of scale-growth on cylindrical samples of iron are essentially the same as those required for the plane problem, but in this case they are more difficult to attain in practice. It is assumed that the oxide remains in contact with the iron everywhere, that it is pure FeO with a negligibly thin layer of the higher oxides maintaining the oxygen-rich wüstite composition at a constant level, and that porosity and end effects can be neglected.

The following quantities will be defined at the outset:

D is the average diffusion coefficient in cm^2-sec^{-1},

c_1 and R_1 are the iron concentration (gm-cm^{-3}) and radius (cm) at the wüstite-iron interface,

c_2 and R_2 are the like quantities at the outer wüstite interface, and

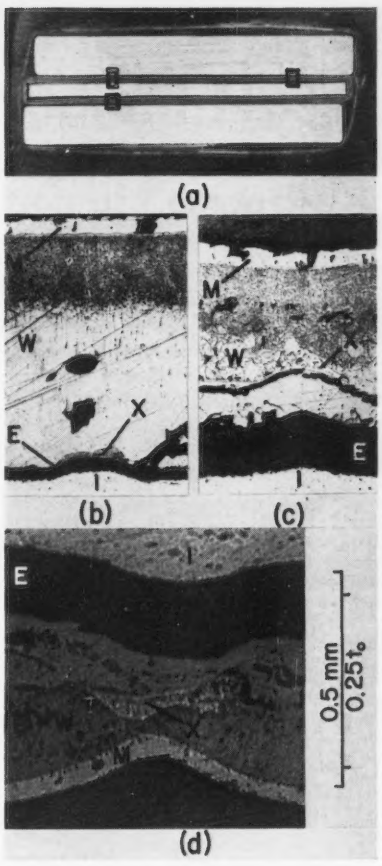

Figure 13—Partially detached scale on a flat Puron specimen. Originally 1/16 inch thick (t_0) and 1 inch long, the blank was oxidized to 13.8 percent oxygen by weight at 900 C.

(a) Sample mounted edgewise in resin between iron blocks and ground to reveal a longitudinal cross-section. The scale has become detached at lower left and upper right. Outlined areas are shown as photomicrographs in (b), (c), and (d) with the same orientations relative to the full photograph.

(b) Inert marker (X) at iron-wustite interface in adherent region of the scale.

(c) Inert marker with wustite overlay at separated region of the scale. The fine structure just below the sharply defined magnetite-wustite boundary here and in picture (b) is precipitated magnetite. Same magnification as in (b).

(d) Inert marker with wustite overlay at the separated region of the scale on the side of the sample opposite to (b). Stannous chloride etch. Enlarged from a low-magnification photograph to a magnification of about 63X, corresponding to that of (b) and (c).

c_0 and R_0 are the density of iron and the original radius of the sample.

At steady state, the iron concentration $c(r)$ within the wüstite layer has no explicit time dependence, so that

$$D\nabla^2 c = 0,$$

whence

$$c = C \ln r + C'.$$

The integration constants C and C′ can be evaluated from the boundary conditions at R_1 and R_2, giving

$$c = \frac{(c_1 - c_2)\ln r}{\ln (R_1/R_2)} + \frac{c_2 \ln R_1 - c_1 \ln R_2}{\ln (R_1/R_2)} \qquad (1)$$

and

$$\nabla c = \frac{C}{r} = \frac{c_1 - c_2}{r \ln (R_1/R_2)} \qquad (2)$$

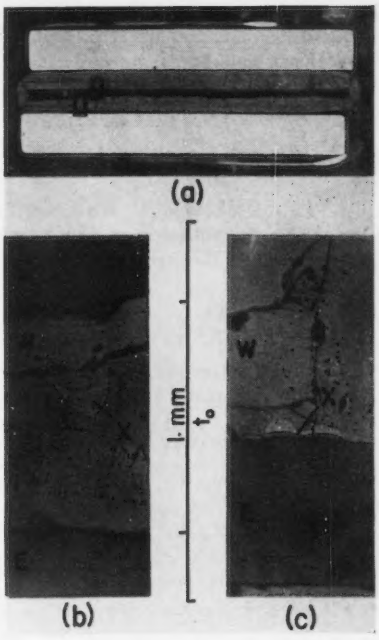

Figure 14—Flat Puron specimen, oxidized to 24.0 percent oxygen by weight and homogenized in argon for 62 hours, at 900 C; the original thickness (t_0) and length were 1/16 inch and 1 inch.

(a) Sample mounted edgewise in resin between iron blocks and ground to reveal a longitudinal cross-section. Outlined areas are shown in photomicrographs (b) and (c) with the same orientations relative to the full photograph.

(b) Inert marker (X) at iron-wustite interface in outside of the scale is toward the bottom of the picture.

(c) Possible position of inert marker near the interior surface of the scale, with slight wustite overlay. No free iron is present, and except for small magnetite deposits near the corners (not visible in this figure), the scale is entirely wustite (W). Stannous chloride etch. Original magnification, 45X.

The condition of conservation of iron provides the relation

$$c_0 R_0^2 = c_0 R_1^2 + 2\int_{R_1}^{R_2} r\, c\,(r)\, dr,$$

which can be evaluated with the aid of (1):

$$c_0 R_0^2 = (c_0 - c_1) R_1^2 + c_2 R_2^2 + \frac{(c_1 - c_2)(R_1^2 - R_2^2)}{\ln (R_1/R_2)^2}. \qquad (3)$$

This can be simplified by letting

$$\gamma = (R_1/R_2)^2 \text{ and } \alpha = (R_1/R_0)^2,$$

which puts (3) into the form

$$c_0 \gamma/\alpha = c_2 + (c_0 - c_1)\gamma - (c_2 - c_1)(\gamma - 1)/\ln \gamma.$$

Now the function $(\gamma - 1)/\ln \gamma$ varies monotonically from 0 to 1 with values of γ between these limits; since $(c_2 - c_1)/(c_0 - c_1)$ is less than 0.03 in the case of wüstite, the value of γ/α will not be altered by more than 1 percent if the function is replaced by γ itself. Doing so, one gets

$$c_0 \gamma/\alpha = c_2 + (c_0 - c_2)\gamma,$$

or

$$\gamma = (1 - \lambda)\alpha/(1 - \lambda\alpha), \qquad (4)$$

where $\lambda = (c_0 - c_2)/c_0$.

Since R_1 and R_2 are now interdependent, one needs only to find the time-

dependence of one of them. To do so, the diffusion equation may be applied to the mass flow at the iron interface:

$$2\pi R_1 L (c_0 - c_1)\, d R_1 = 2\pi R_1 L D (\nabla c)_{R_1}\, dt$$

$$= 2\pi R_1 L \frac{D (c_1 - c_2)}{R_1 \ln (R_1/R_2)}\, dt,$$

where L is the length of the sample. Introducing the values of α and γ as given above into this relation one finds that

$$\ln \gamma\, d\alpha = \frac{4 D (c_1 - c_2)}{R_0^2 (c_0 - c_1)}\, dt.$$

This can be integrated over α from 1 to α and over t from 0 to t, yielding

$$\frac{4 D (c_1 - c_2)}{R_0^2 (c_0 - c_1)}\, t = \frac{(1 - \lambda\alpha)}{\lambda} \ln \frac{1 - \lambda\alpha}{1 - \lambda} + \alpha \ln \alpha. \quad (5)$$

If one now takes the square root of (5) and introduces K_p, the parabolic rate constant conventional in plane geometric considerations, one can write the form

$$\frac{K_p}{R_0} t^{1/2} = k_0 \left\{ \frac{(1 - \lambda\alpha)}{\lambda} \ln \left(\frac{1 - \lambda\alpha}{1 - \lambda} \right) + \alpha \ln \alpha \right\}^{1/2}, \quad (6)$$

where the constant k_0 is yet to be determined.

It is now necessary to express the mass increase of the sample in terms of α. If ξ is the average oxygen concentration in wüstite, this is simply

$$\Delta M = \xi \pi (R_2^2 - R_1^2) L.$$

Using the definitions of α and γ and their inter-relation as given in (4), this is

$$\Delta M = \xi \pi R_0^2 L (1 - \alpha)/(1 - \lambda),$$

so

$$\Delta M/(R_0 A_0) = \frac{1}{2} \xi (1 - \alpha)/(1 - \lambda), \quad (7)$$

where A_0 is the initial surface area $2\pi R_0 L$, end effects being neglected.

For very short times, $\alpha \sim 1$, and the parabolic rate law is followed since the thin cylindrical scale approximates a plane lamina.

Therefore,

$$\lim_{\alpha \to 1} \left\{ \left[\Delta M/(R_0 A_0) \right] \Big/ \left[(K_p/R_0)\, t^{1/2} \right] \right\} = \xi \left[2 k_0^2 (1 - \lambda) \right]^{-1/2} = 1$$

and

$$k_0 = \xi \left[2 (1 - \lambda) \right]^{-1/2}.$$

Replacing this in (6),

$$\frac{K_p}{R_0} t^{1/2} = \frac{\xi}{(2\lambda)^{1/2}} \left[\left(\frac{1 - \lambda\alpha}{1 - \lambda} \right) \ln \left(\frac{1 - \lambda\alpha}{1 - \lambda} \right) + \left(\frac{\lambda\alpha}{1 - \lambda} \right) \ln \alpha \right]^{1/2}. \quad (8)$$

Then $\Delta M/(R_0 A_0)$ and $(K_p/R_0)\, t^{1/2}$ are related through the variable α according to (7) and (8), as long as the concentration limits are fixed at some values c_1 and c_2, (i.e., until the free iron supply is entirely depleted). Using average concentration values extracted from the data of Himmel, Mehl, and Birchenall,[2] namely,

$$c_0 = 7.86 \text{ gm-cm}^{-3},$$

$$c_2 = 4.30 \text{ gm-cm}^{-3},$$

and

$$\xi = 1.33 \text{ gm-cm}^{-3},$$

one can write (7) and (8) in numerical fashion as follows:

$$\Delta M/(R_0 A_0) = 1.215 (1 - \alpha); \quad (9a)$$

$$[\Delta M/(R_0 A_0)]_{max} = 1.215 \text{ gm-cm}^{-3}; \quad (9b)$$

$$(K_p/R_0)\, t^{1/2} = 1.397 \{1.828 (1 - 0.453\alpha) \ln [1.828 (1 - 0.453\alpha)] - 0.828 \alpha \ln \alpha\}^{1/2}; \quad (10a)$$

$$[(K_p/R_0)\, t^{1/2}]_{max} = 1.468 \text{ gm-cm}^{-3}; \quad (10b)$$

in which $\alpha = (R_1/R_0)^2$. The thickness d of the wüstite layer, incidentally, is given by

$$d/R_0 = (2\Delta M)/(\xi R_0 A_0) = 1.82 (1 - \alpha),$$

the maximum being

$$[d/R_0]_{max} = 1.82.$$

Plots of (9a) versus (10a) are shown as dashed lines in Figures 4, 6, 8, and 10.

Any discussions of this article not published above will appear in the June, 1958 issue

Corrosion and Metal Transport In Fused Sodium Hydroxide*

Part 3—Formation of Composite Scales on Inconel

By G. PEDRO SMITH,* MARK E. STEIDLITZ* and EUGENE E. HOFFMAN*

Abstract

Inconel was exposed to fused sodium hydroxide at temperatures of 450 to 815 C and for times up to 400 hours. The observed reaction was shown to consist in the selective leaching of iron and chromium from their solid solution with nickel. The reaction product, a mixture of oxides and sodium oxysalts, was found to grow in a network of channels which, starting from the metal surface, penetrated into the bulk Inconel. The geometry of this network differed considerably with different experimental conditions and is described in detail. Rate measurements were made and the results reported. 4.7

Introduction

THE CORROSION mechanism of alloys at high temperatures is complicated by diffusional processes within the metal. This is especially true if different alloy components which have markedly different chemical reactivities are used. One of these complexities is the formation of two-phase corrosion product layers which consist of a mixture of reaction product and of metal enriched in the more reaction-resistant constituents. This type of corrosion is well illustrated by the reaction between fused sodium hydroxide and Inconel (nominal composition: 78 percent Ni, 15 percent Cr, and 7 percent Fe), which is described in this paper.

In a cursory study of the action of sodium hydroxide on alloys, Craighead, Smith, and Jaffee[1] found that Inconel was only slightly attacked in 24 hours at 538 C but that at 815 C a sheet of unspecified thickness was completely consumed.

Of the primary constituents of Inconel, elemental nickel is known to be very corrosion resistant in fused sodium hydroxide up to 815 C, whereas iron and chromium react rapidly at temperatures well below 815 C.[1,2]

General Procedure

Most of the hydroxide-Inconel reactions were carried out by the "controlled-atmosphere test" procedure described in a previous paper.[3] Tests were conducted at temperatures of 450 to 800 C for times up to 400 hours under blanketing atmospheres of purified hydrogen and purified helium. A few tests were made by the "capsule test" technique at 815 C on specimens which had been stressed by cold rolling. These stressed specimens were not annealed before they were tested.

The metal used for most tests was commercial Inconel, but representative tests were conducted with an alloy which had been vacuum-melted and cast from the elemental metals and which had an analyzed composition of 78.2 percent Ni, 14.17 percent Cr, 7.30 percent Fe, 0.14 percent Si, 0.10 percent Cu, 0.10 percent Mn, 0.02 percent Al, 0.13 percent Mg, 0.008 percent C, 0.003 percent S, and less than 0.0003 percent Ti. This alloy, although purer than commercial Inconel, did not appear to differ from the commercial material in its corrosion behavior.

Determinations were made of microstructures, rates of corrosion, and chemical constitution of corrosion products as described below. Metallographic specimens were prepared by standard procedures except that a special effort had to be made to minimize exposure of polished specimens to moist air, since the corrosion products were hygroscopic. In this paper all photomicrographs show sections perpendicular to the specimen-hydroxide interface in the as-polished condition. The surface which was exposed to attack is toward the top of each picture.

Microstructures

For the range of experimental conditions studied in this research the corrosion of Inconel always took place by the formation of two-phase regions. Other forms of corrosion such as pitting, uniform dissolution of surface layers, and formation of uniform surface layers of oxides or oxysalts were negligible. The microstructures of the two-phase corrosion-product regions were found to differ considerably with differences in experimental conditions.

Corrosion Under Hydrogen

The corrosion product microstructures formed when Inconel was exposed to fused sodium hydroxide under a blanketing atmosphere of hydrogen differed from those formed under other test conditions. In samples corroded at 650 C and below for times up to 400 hours, two-phase corrosion-product regions were confined largely to intergranular boundaries and adjacent regions. The faint gray lines forming a network near the surface of the specimen shown in Figure 1 are regions where particles of the nonmetallic phase occurred most densely in a specimen corroded for 100 hours at 600 C. In Figure 2 individual particles of the nonmetallic phase may be distinguished. These particles appear as long, slender spines. Most of the spines were confined to the immediate neighborhood of grain boundaries, but some extended far into the interior of metal grains with an apparent preference for specific crystal orientations of the metal matrix. The spines were anisotropic toward reflected, plane-polarized light and showed two light extinctions when the microscope stage was rotated. From this it was concluded that the spines were single crystals.[4] Figure 2 was taken with the aid of polarized light in order to obtain a contrast between the metallic and nonmetallic phases.

At 700 and 800 C, nonmetallic particle formation was sometimes initiated at grain boundaries, but within less than 10 hours the formation spread throughout the grains at the surface and thereafter continued in the form of a thick-

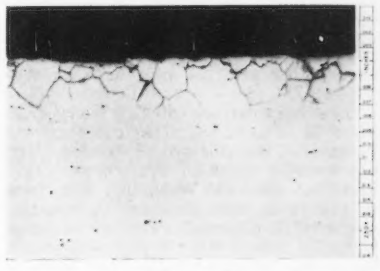

Figure 1—Intergranular attack on Inconel after 100-hour exposure to NaOH at 600 C under hydrogen. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

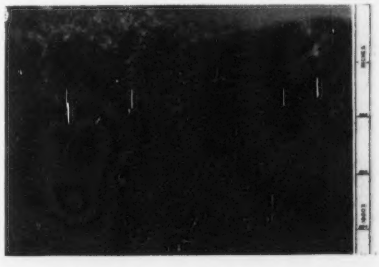

Figure 2—Nonmetallic crystals formed at intergranular regions on Inconel after 100-hour exposure to NaOH at 600 C under hydrogen. Sample was photographed with the aid of reflected, plane-polarized light; analyzer and polarizer were in crossed positions. Unetched. Original magnification, 1000X; actual magnification after reduction for engraving purposes, 330X.

★ Submitted for publication June 26, 1957. Part 1 of this series appeared in the September, 1957 *Corrosion*, page 561t; Part 2 appeared in the October, 1957 issue, page 627t.

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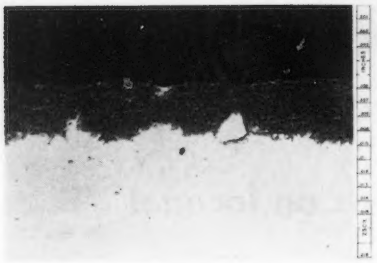

Figure 3—Two-phase corrosion product layer formed on Inconel after 100-hour exposure to NaOH at 700 C under hydrogen. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

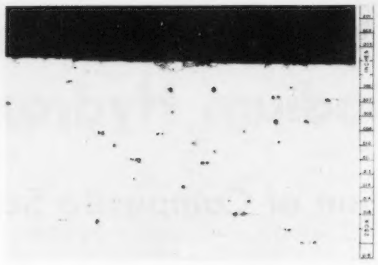

Figure 6—Intergranular attack on Inconel after 6-hour exposure to NaOH at 700 C under helium. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

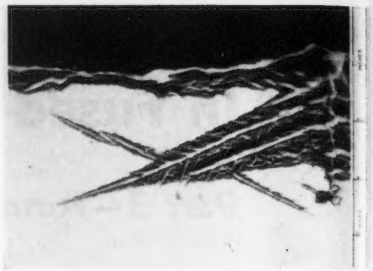

Figure 8—Wedge-shaped structure formed on Inconel after 100-hour exposure to NaOH at 600 C under helium. Unetched. Original magnification, 1000X; actual magnification after reduction for engraving purposes, 330X.

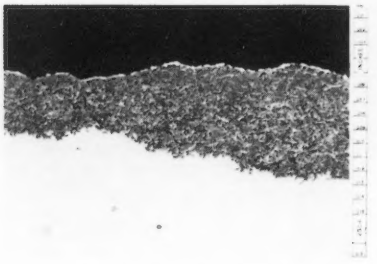

Figure 4—Two-phase corrosion product layer formed on Inconel after 100-hour exposure to NaOH at 800 C under hydrogen. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

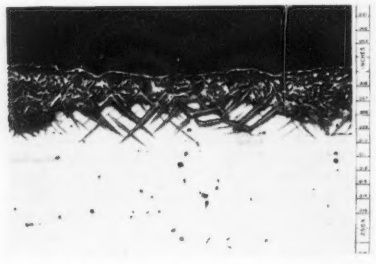

Figure 7—Two-phase corrosion product layer formed on Inconel after 47-hour exposure to NaOH at 700 C under helium. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

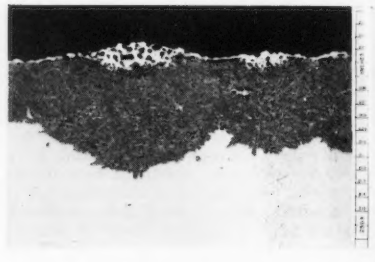

Figure 9—Two-phase corrosion product layer formed on Inconel after 100-hour exposure to NaOH at 800 C under helium. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

Figure 5—Microstructure within a two-phase region formed on Inconel after 100-hour exposure to NaOH at 800 C under hydrogen. Unetched. Original magnification, 1000X; actual magnification after reduction for engraving purposes, 330X.

ening layer of two-phase corrosion product. Such layers are seen in Figures 3 and 4, which show corrosion after 100 hours at 700 and 800 C, respectively. The distribution density of nonmetallic particles was less at 700 than at 800 C. At 800 C some of the nonmetallic particles occurred as relatively large, irregular masses. Figure 5 shows a portion of a two-phase corrosion-product film which formed during a 100-hour test at 800 C. This figure is at a sufficient magnification for some of the details of particle size and distribution to be resolved.

In most tests the two-phase corrosion product films which formed at 700 and 800 C were not uniform in thickness. Two of the most common nonuniformities, illustrated in Figures 3 and 4, were: (1) unattacked patches of metal of about the dimensions of grains in the base metal, shown in Figure 3; (2) much more gradual variations in film thickness, shown in Figure 4.

At 700 and 800 C the two-phase films advanced somewhat more rapidly along intergranular regions than through grains. This intergranular penetration was never pronounced but was frequently greater than is illustrated by the figures which accompany this paper.

Corrosion Under Helium

When corrosion took place under helium rather than hydrogen, several significant differences were found for the geometric shapes of two-phase regions. The two-phase product was confined to the neighborhood of grain boundaries for exposures of less than 10 hours at temperatures up to 800 C. Most of the nonmetallic crystals lay in two layers, one on either side of the grain boundary, with a metallic layer in between. Figure 6 shows a microstructure of this sort. Here the reaction was allowed to proceed for only 6 hours; therefore the attack was shallow and was confined to a few patches at the surface.

When the reaction was allowed to continue for more than 10 hours at a temperature between 600 and 725 C, wedge-shaped regions of two-phase product extended from near the hydroxide-Inconel interface for some distance into the underlying metal. With increasing time, such wedge-shaped structures became interconnected to form a continuous two-phase film at the specimen surface with wedges projecting beneath the film as shown in Figure 7.

The substructure of the wedges, as seen in Figure 8, was complex but regular. A metallic core extended along the median plane of the wedge. Small metallic dendrites branched out from this core at approximately a constant angle. Other metallic regions of more irregular shapes were scattered throughout each wedge. Near the tip of a wedge, slender spine-like crystals of the nonmetallic phase extended out from a narrow metallic core.

Within the base-film from which the wedges grew, there was no observable geometric regularity except that the metallic cores of many wedges could be traced back for some distance into this region (see Figure 7).

At 800 C the two-phase region occurred as a fine-grained layer at the surface of the Inconel, as shown in Figure 9. In many ways it resembled the layers which formed under similar conditions of time and temperature with a blanketing atmosphere of hydrogen, but there was one notable difference: under helium, strings of relatively large, nonmetallic particles occurred which ran in almost straight lines for considerable distances through, and protruded somewhat below, the two-phase layers. Such strings are faintly visible in Figure 9 and are seen more clearly in Figure 10, which shows a part of the lower boundary of a two-phase region.

Grain-Coarsening Phenomena

The surface layers of the two-phase corrosion product which formed under helium frequently had a very coarse grain size. Some regions of coarsened material are evident at the surface of the specimen shown in Figure 9. Figure 11 shows an unusually thick layer of coarse-grained corrosion product. The sponge-like structure of the metallic phase shown in this figure was typical of all but the thinnest coarse-grained layers when corrosion took place at 800 C.

The transition from fine-grained to coarse-grained material was quite abrupt, as can be seen in Figure 11. However, examination of Figure 12 revealed a narrow transition zone, within which most of the slender spine-like crystals of nonmetallic phase disappeared and collected into much larger masses. The metallic phase collected into interconnected regions which had

Figure 10—Microstructure at lower boundary of a two-phase region formed on Inconel after 100-hour exposure to NaOH at 800 C under helium. Unetched. Original magnification, 1000X; actual magnification after reduction for engraving purposes, 330X.

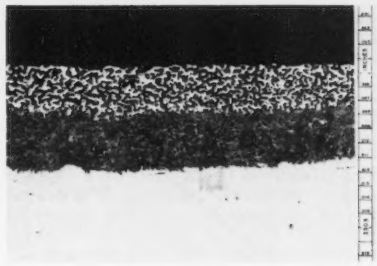

Figure 11—Photomicrograph showing coarse-grained region in upper part of corrosion product layer formed on Inconel after 100-hour exposure to NaOH at 800 C under helium. Unetched. Original magnification, 250X; actual magnification after reduction for engraving purposes, 83X.

Figure 12—Photomicrograph showing transition from fine-grained to coarse-grained structure in corrosion product formed on Inconel after 100-hour exposure to NaOH at 800 C under helium. Unetched. Original magnification, 1000X; actual magnification after reduction for engraving purposes, 330X.

TABLE 1—Corrosion Data for Five Runs at 800 C for 100 Hours*

Run No.	Gas	No. of Meas.	Av. Corr.	Max. Corr.	Min. Corr.	Max. Av.	Min. Av.
68	H_2	23	9.0	12.0	3.6	11.0	5.0
71	H_2	22	5.7	10.6	0.0**	9.8	3.2
91	He	20	7.1	9.4	3.6	8.0	6.6
92	He	12	6.4	9.0	2.0	7.4	4.2
95	He	14	7.5	9.8	3.2	8.7	6.6

* Numerical values in mils.
** Next largest values: 0.6, 0.6, and 1.0.

many of the geometric characteristics of a partly sintered mass of metallic particles. A few, small, nonmetallic particles appeared to have remained trapped within the metallic phase.

Under an atmosphere of hydrogen, and at temperatures of 600 and 700 C under helium, a thin discontinuous layer of metal often occurred at the surface of the corrosion product film, as may be seen in Figures 4, 7 and 8. At a high magnification it was found that there was a transition from the two-phase corrosion product layer to the surface film of metal.

Washing with weak acids made it possible to leach a substantial portion of the nonmetallic phase out of the coarse-grained corrosion products. After such leaching, the metal remained a coherent, interconnected structure. The fact that the nonmetallic phase could be leached in this manner showed that it too was largely interconnected. Of course, the nonmetallic phase may have contained a few isolated metal particles, and the metallic phase definitely contained a few, small, nonmetallic particles which could not be leached and which probably were isolated.

Quantitative Corrosion Measurements

The corrosion of Inconel took place largely by intergranular penetration and by the formation of two-phase corrosion product films. Some metal entered the liquid phase as dissolved corrosion products, but this was small compared with the mass of metal affected by internal attack.

Measurements of the amount of corrosion were made with the aid of a Vickers projection microscope. An image of a specimen was projected at an accurately determined magnification of 250, and measurements were made of the image with calibrated scales to within 0.2 mil (1 mil = 0.001 in.). The maximum and minimum corrosion which was to be found on the entire length of specimen microsection was measured. Then an average corrosion was determined, the method of determination depending upon the kind of microstructure. When corrosion took place by two-phase film formation, the average corrosion was determined as the arithmetic average of the film thickness by measuring the cross-sectional area of corrosion-product film along a 20-mil length of specimen surface and numerically dividing by 20 to give the average thickness for the 20-mil length. Average thicknesses were determined in this way at 10 to 25 places on a specimen, and the resulting values were arithmetically averaged to give an average film thickness for the entire specimen. The arithmetic averaging of values for 20-mil lengths was justified, inasmuch as the individual values generally fell on a symmetrical distribution curve. This rather complicated averaging procedure was necessary because the corrosion product films seldom approached uniform thickness.

Special problems arose in obtaining the average corrosion when the film was studded with wedge-shaped fingers such as those shown in Figure 7. (These films occurred only with a helium atmosphere and for temperatures of 600 to 725 C). For such films the metal regions in between the tips of the wedge-shaped fingers were counted as part of the film. This procedure is subject to errors in judgment on the part of the observer, but film geometry was such that these errors were not great, and a more objective procedure for evaluating average corrosion was considered prohibitively time-consuming.

These film thickness measurements did not give directly the depth of metal penetrated by corrosive attack since the films occupied appreciably more volume than that of the metal consumed. In order to determine this volume increase, specimens of accurately known thicknesses were corroded for 100 hours at 800 C under hydrogen. After corrosion, the film thickness and total specimen thickness were measured at a number of sites. The films were found to be approximately 30 percent thicker than the depth of metal which had been corroded.

Average intergranular penetration was measured on 20-mil lengths as the distance from the surface to the mid-point between two lines which were parallel to the surface and which included between them the termini of all corroded boundaries along the 20-mil length. An aspect of intergranular corrosion which was not quantitatively considered in this work was the fraction of available grain boundaries actually attacked. Qualitatively, it was observed that when grain-boundary penetration was small, only a portion of the available boundaries was attacked but that as the penetration went deeper a much greater portion was attacked. This will be seen in comparing Figure 1 with Figure 6.

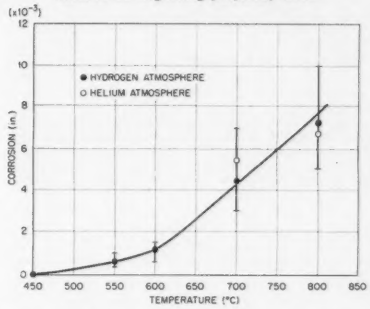

Figure 13—Summary of values of average corrosion after 100 hours. Values are plotted only for conditions under which at least three tests were run. Vertical bars show the scatter of values for tests under a hydrogen atmosphere.

A compilation of all average corrosion values for conditions under which at least triplicate runs were made is presented in Figure 13. This plot shows the average corrosion as a function of temperature for 100-hour periods. The values at 550 and 600 C are for grain-boundary corrosion, and the values at 700 and 800 C are for film formation. The open and closed circles represent values obtained by averaging the average corrosion for all runs at a given temperature and under the specified atmosphere. The average corrosion for individual runs scattered considerably about this over-all average. The range of scatter for runs under hydrogen is denoted by a vertical line at each temperature. The range of scatter for runs under helium was about the same as that for hydrogen. Two conclusions are drawn from Figure 13. First, the corrosion rate accelerated with temperature as would be expected for any activated process. Second, corrosion under hydrogen and under helium did not differ significantly in extent at 700 and 800 C, the only temperatures for which comparable values were obtained.

Figure 13, of course, tells nothing of the variation of corrosion from place to place on individual specimens. Some

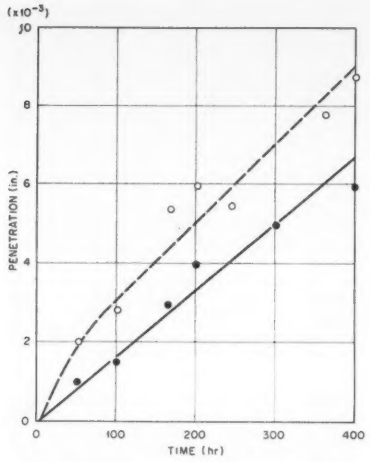

Figure 14—Intergranular penetration of Inconel exposed to NaOH at 650 C under hydrogen as a function of time. The closed circles and solid curve represent average penetration, whereas the open circles and dotted curve represent maximum penetration.

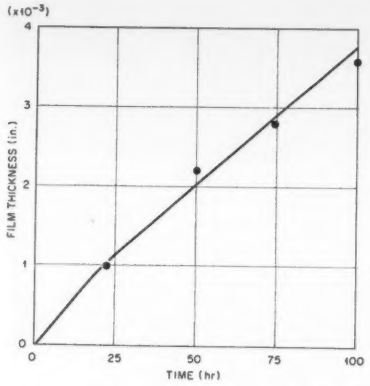

Figure 15—Corrosion product film thickness on Inconel exposed to NaOH at 700 C under hydrogen as a function of time.

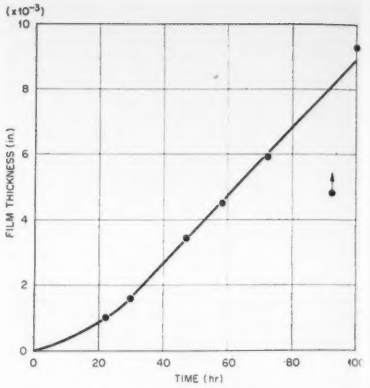

Figure 16—Corrosion product film thickness on Inconel exposed to NaOH at 725 C under helium as a function of time.

concept of these variations may be obtained from Table 1, which gives values obtained from five runs of 100 hours each at 800 C, two under hydrogen and three under helium. These runs were selected as representative of a total of five runs under hydrogen and of ten under helium. The columns headed "Max. Corr." and "Min. Corr." give the greatest and the least corrosion observed anywhere on each specimen. Although these extreme values deviate significantly from the average, they generally were associated with quite localized conditions such as are illustrated by the specimen in Figure 3. However, if there was an extreme deviation at one site on a metal, there were usually several others which deviated almost as much. For example, it was surprising to find a small patch of surface on the specimen from Run 171, which had suffered no observable corrosion. However, this was not entirely unique, since there were a number of patches with almost no corrosion, as is indicated in the footnote to Table 1.

The columns headed "Max. Av." and "Min. Av." give, respectively, the greatest and least average corrosion found on individual 20-mil lengths. The deviations of these values from the average corrosion give a measure of the prevalence of variations of film thickness over the specimen surface. Some runs, such as No. 191, showed relatively small variations in thickness, while others, such as No. 171, showed quite wide variations.

Figures 14, 15, and 16 show corrosion as a function of time at constant temperature for three different corrosion-product microstructures. These curves were obtained by simultaneously corroding a number of specimens in the same hydroxide melt and then at specified times withdrawing individual specimens. These specimens were withdrawn by means of a wire passing through a vacuum-tight gland, so that they would be in a cool portion of the controlled-atmosphere apparatus. Individual points in Figures 14, 15, and 16 represent averages taken over individual specimens. All the points on any one figure represent averages from specimens corroded together in the same melt for overlapping intervals of time.

Figure 14 gives corrosion data for Inconel exposed to sodium hydroxide under hydrogen at 650 C for times up to 400 hours. The corrosion consisted entirely of grain-boundary penetration. The closed circles and the solid curve represent values of average penetration for each specimen, while the open circles and the dashed curve represent values of maximum penetration. The trend of both curves is essentially the same.

Figure 15 gives the average film thickness for specimens exposed for various times up to 100 hours at 700 C under hydrogen. Films which looked like the film shown in Figure 3 were formed on all specimens.

Figure 16 gives the average film thickness for specimens exposed for various times up to 100 hours at 725 C under helium. The corrosion product films had many wedge-shaped fingers penetrating into the substrate metal and looked much like the specimen shown in Figure 7. Most of the points lie on a fairly smooth curve except for the point at 92 hours, which is entirely out of line. The origin of this discrepancy is not known. The rate of corrosive penetration at a given time may be obtained from the slope of a penetration versus time curve such as the curves in Figures 14, 15, and 16. These latter curves did not deviate greatly from linearity except for a brief initial transient.

A large number of measurements were made of the thicknesses of coarse-grained layers at the surfaces of corrosion-product films which formed at 800 C. (Such a coarse-grained layer is shown in Figure 11.) Under helium these layers averaged 10 to 20 percent and reached a maximum of about 50 percent of the total corrosion-product thickness. For a hydrogen atmosphere the layers averaged 5 to 10 percent and reached a maximum of about 20 percent. There was, however, a large variation in the relative thickness of these layers from specimen to specimen and between different places at the surface of any one specimen. In some specimens the coarse-grained layer was entirely absent, in some it consisted of small patches scattered over the surface, and in others it occurred as a layer of uniform thickness.

Corrosion Product Analysis

Several analytical procedures were used in an effort to obtain information on the chemical constitution of the two-phase corrosion products. All measurements were made on specimens which had been corroded for 100 hours at 800 C.

Samples of corrosion product from which a substantial portion of the nonmetallic constituent had been leached were examined by x-ray diffraction techniques. The predominant phase was a face-centered cubic structure with a lattice constant of (3.5207 ± 0.0002) kX units, which obviously represents slightly contaminated nickel.

Rees, Burns, and Cook[8] made an x-ray study of the nickel-iron-chromium system. According to their data, the above lattice constant corresponds to nickel containing about 3 wt percent iron plus chromium in solid solution. This depletion of iron and chromium in Inconel on reaction with fused sodium hydroxide is in agreement with the fact that iron and chromium react fairly readily with a hydroxide melt, whereas nickel does not. Consequently, the metallic constituent in the two-phase corrosion product is taken to be nickel containing a small percent of iron plus chromium.

Corrosion-product samples were strongly ferromagnetic, as would be expected from the composition of the metallic phase. Cross sections of corroded Inconel were metallographically polished, covered with a thin layer of an aqueous suspension of magnetic iron oxide, and microscopically examined while a magnetic field was applied. The particles of suspended oxide migrated quickly and exclusively to the two-phase corrosion-product layer.

Samples of corrosion product from several experiments were mechanically spalled away from the unattacked substrate and analyzed for nickel, chromium, iron, and sodium. Sodium was determined by flame photometry and the other three metals by volumetric methods. The analyses did not vary much among samples, and the averages of the results, together with the maximum deviation among samples, were as follows: 63.6 ± 0.3 wt percent Ni; 13.2 ± 0.3 wt percent Cr; 5.9 ± 0.1 wt percent Fe; and 6.4 ± 0.1 wt percent Na.

Effect of Cold Work

Tests were conducted on specimens of Inconel which had been cold-rolled by various amounts up to a 50 percent reduction in thickness. These specimens were allowed to react with sodium hydroxide for 8 hours at 815 C. The resultant corrosion-product microstructures were essentially the same as those found for corrosion at high temperatures in short-time tests. That is, attack was primarily along intergranular boundaries with a distribution density of transgranular needles of inorganic compound which was high near the hydroxide-metal interface but which decreased rapidly away from this interface.

The effect of cold work on this corrosion-product microstructure was twofold. First, the depth of penetration increased in a regular way with increasing prior cold work up to a penetration which, for 50 percent cold work, averaged twice that for no prior cold work. Second, the distribution density of transgranular reaction increased with increasing prior cold work.

Discussion

It was shown that the primary manifestation of the reaction of Inconel and sodium hydroxide was the formation of a corrosion product which consisted of a metallic and a nonmetallic phase. The metallic phase was found to be nickel containing a small amount of iron and chromium in solid solution. A definitive determination of the nonmetallic phase was not made, but enough data were obtained to justify a few conclusions concerning its chemical nature.

The weight ratio of nickel to chromium to iron in the two-phase regions was found to be 1.00:0.208:0.0928. This is very close to the corresponding ratio for unattacked Inconel. Consequently, the iron and chromium, which were much depleted in the metallic phase, must have been primary constituents of the nonmetallic phase. No information was obtained to indicate whether or not nickel was an important constituent of the nonmetallic phase, although from the amount of nickel present in the metallic phase, it is obvious that only a small part of the nickel which could have reacted actually did so. The total quantity of nickel, chromium, iron, and sodium present in the two-phase region was 89 wt percent. Since these were the only metallic elements present in significant quantity, the undetermined 11 wt percent must have consisted of the nonmetallic elements oxygen and hydrogen.

The mole ratio of nickel to chromium to iron to sodium in the corrosion product layer was 1.00:0.234:0.097:0.258; that is, there were more moles of sodium than either chromium or iron. This high sodium ion content shows that the corrosion product contained a significant quantity of either sodium hydroxide or of sodium oxide. The latter, if present, probably was combined with the heavy metal oxides to form sodium oxysalts.

The above facts, when coupled with a knowledge of the reaction products of elemental metals and fused sodium hydroxide,[3] suggest rather strongly that the nonmetallic phase consisted of oxides and sodium oxysalts of chromium and iron with or without a certain quantity of nickel ions. This postulate is quantitatively reasonable provided that the undetermined 11 percent of the weight of the two-phase region is assigned largely to oxide ions.

The nonmetallic phase clearly corresponded to the chemical compounds which resulted from the Inconel-sodium hydroxide reaction, while the metallic phase was that part of the Inconel which did not react. Thus the over-all chemical reaction consisted in the selective leaching of iron and chromium from solid solution, possibly together with some nickel, leaving behind most of the nickel unreacted.

The experiments in which the nonmetallic component was separated from the coarse-grained corrosion product showed that, for this microstructure at least, the nonmetallic component formed an interconnected network which was in contact with the fused hydroxide. It is probable that the fine-grained product also formed a network which was in contact with the fused hydroxide; otherwise it is difficult to account for the presence of considerable quantities of sodium ions in the two-phase region. It is concluded, therefore, that each observed microstructure consisted of a cross section through a network of oxide-oxysalt compounds which extended continuously from the hydroxide-Inconel interface throughout the corroded region.

Measurements were made of the depth of corrosive penetration as a function of time. These measurements gave the rate of advance of the tips of the corrosion-product network. (It is important to recognize that this rate of advance is not to be equated to the over-all rate of reaction, concerning which no measurements were made, because it is not improbable that lateral growth of the network continued at points removed from the advancing tips.) The data obtained showed that the rate of advance did not change with time after an initial transient of a few hours' duration. This fact suggests that transport processes through the corrosion-product layer restricted the rate of advance only in a secondary way. Hence, the more important rate-determining steps must have been localized at configurations which did not change in a significant way with time. Two such configurations were the corrosion product-hydroxide melt interface and the ends of the advancing network. Of these two, the network ends seem the more plausible choice, partly because most processes at the corrosion product-hydroxide melt interface, such as diffusion through the lamellar layer of liquid, would be expected to be fast processes, and partly because prior cold work was shown to accelerate the rate of advance.

Wagner[6] pointed out that when the rate of displacement of a boundary between two phases is controlled by diffusion of a reactant through the phase whose volume decreases, there is a tendency for the advancing interface to buckle at localized sites and to protrude into the latter phase. Applied to the reaction between Inconel and sodium hydroxide, this mechanism suggests that the reason for the reaction product growing in a network of channels rather than in a uniform film on the metal surface is that the rate of reaction was controlled by the rate of diffusion of iron and chromium through Inconel rather than by rates of diffusion through the reaction-product network. Of course, as Wagner points out, the rate of diffusion through the reaction product will exert a secondary influence on the over-all reaction rate because here a series-diffusion process is being dealt with. This mechanism is in general agreement with the rate data presented in this paper.

Measurements of the effect of prior cold work showed that stressed Inconel is more susceptible to attack than unstressed Inconel. This suggests that those stresses which undoubtedly developed by expansion processes in the corrosion-product layer may have played an important role in corrosion. The growth of the corrosion-product layer was accompanied by a volume expansion of about 30 percent. This expansion must have been localized in the inorganic reaction product as distinguished from the leached alloy and, hence, represented a localized expansion very much greater than 30 percent. The advancing front of the reaction-product network always consisted of slender shapes which penetrated into the unattacked metal. Under a blanketing atmosphere of hydrogen these shapes were thin wedges advancing along intergranular boundaries from which branched long acicular crystals into the transgranular regions. Under a blanketing atmosphere of helium the slender shapes consisted of wedges, the tips of which were studded with acicular particles. Since the oxide phase occupied a significantly greater volume than that of the metal removed by leaching, there must have been a substantial component of tensile stress normal to the axes of the slender, penetrating particles. Mears, Brown, and Dix[7] have pointed out that such a stress would create a stress concentration at the base of the localized corrosion path. At elevated temperatures, metal will plastically deform and relieve such stresses. However, over the range of temperatures considered in this report, stresses are relieved in Inconel at a rate which is very much slower than the rate of advance of the corrosion product. Thus the slender particles are envisioned as self-generating a stress at their growing tips.

The experimental measurements showed that on a macroscopic scale the rate of leaching is increased by stresses, and it is postulated that a similar effect is operative in the microscopic region of stress concentration at growing network tips.

The grain coarsening which took place at the surfaces of the two-phase corrosion-product layers can be explained in terms of a particle growth under the influence of a tendency to decrease interfacial free energy by decreasing the interfacial area.

Conclusions

The observed reaction between Inconel and fused sodium hydroxide consisted in the selective leaching of iron and chromium from their solid solution with nickel. The reaction product, a mixture of oxides and sodium oxysalts, grew in a network of narrow channels which, starting from the hydroxide-metal interface, penetrated along preferred paths into the underlying Inconel.

These paths consisted of intergranular boundaries and selected crystallographic directions in the transgranular regions.

The rate of advance of the corrosion-product network did not change much with increasing depth of penetration; therefore the rate-controlling process is envisioned as being localized at the advancing ends of the network, with transport processes along the network exerting only a secondary influence.

The channels created by the leaching process were not large enough to accommodate the volume required for the reaction product. Therefore considerable stresses occurred and were localized at the advancing tips of the reaction-product network. Inasmuch as residual stresses from prior cold work were found to increase the susceptibility of Inconel to attack, it is proposed that the self-generated stresses at advancing ends of the reaction-product network played an important role in the rate-controlling process.

In two-phase corrosion product regions where reaction had substantially subsided, a grain coarsening occurred which probably took place under the influence of a decrease in interfacial free energy.

Acknowledgment

The authors were assisted by R. J. Gray and N. M. Atchley, who prepared the metallographic specimens and took the photomicrographs, and by H. L. Yakel and R. M. Steele, who did the x-ray work.

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Any discussions of this article not published above will appear in the June, 1958 issue

Structural Features of Corrosion Of Aluminum Alloys in Water at 300 C*

By KURT M. CARLSEN

Abstract

A number of aluminum alloy specimens were immersed in distilled water heated to 300 C and a study made of the structural features of corrosion. Samples were of the following general types: Al-Ni alloys, Al-Fe alloys, Al-Ni-Fe alloys, Al-Cu alloys and Al-Ni-Si alloys. After exposure samples were examined by means of a metallographic microscope. In a number of cases the specimens were ground on a metallographic polishing wheel to reveal structural details of the corrosion process. Photomicrographs of both polished and unpolished specimens are included with the paper. 6.4.2

Introduction

DRALEY AND RUTHER[1] have shown that aluminum alloys containing cathodic elements have good aqueous corrosion resistance at high temperatures. Such alloys, while free from a tendency to sudden breakdown, still have corrosion rates which are fairly high compared to those for zirconium alloys. The present study was undertaken to provide some information on the factors determining this rate of corrosion.

Experimental

Alloys were made from ingots of 99.99 percent pure aluminum (supplied by A/S Vigelands Brug), high conductivity copper, electrolytic nickel (99.98 percent), electrolytic iron (British Drug House) and silicon powder (99.5 percent pure, from A/S Bjolvefossen). These ingots were melted in graphite crucibles and allowed to solidify in the crucible. Alloys were used in the as-cast condition, except the Al-Cu alloys which were solution heat treated at the appropriate temperature, and subsequently quenched in water at room temperature.

All specimens were electropolished in a bath containing 100 ml perchloric acid (specific gravity 1.54) and 900 ml alcohol. Test samples were placed in small stainless steel autoclaves, the bottoms of which were covered with high purity aluminum oxide-hydrate to prevent electrical contact between test sample and autoclave. The autoclaves were heated to 300 C in an oven and kept there for the desired time. After exposure, samples were examined by means of a metallographic microscope. Very often only heaps of corrosion products were seen; in such cases the specimen was ground carefully on a metallographic polishing wheel to remove the loose corrosion products and to reveal structural details of the corrosion process.

Water used in all experiments was distilled water.

Results

Al-Ni Alloys

The structure of an as-cast alloy containing 0.92 percent Ni consists, as will be seen from Figure 1, of a $NiAl_3$ eutectic embedded in a matrix of pure Al. Upon short exposures to distilled water at 300 C, one observes preferential corrosion of the eutectic regions, shown by heaps of white corrosion products, while the matrix becomes covered with a thin black and apparently dense layer. Later on part of this layer breaks up and the attack in this part of the matrix becomes more uneven and stronger. The corrosion products in the matrix arising from this second stage give reflection of polarized light when viewed with crossed nickels, while the black layer gives no such reflection. The development can be seen on Figure 2, which shows the breaking up of the black layer as well as heavy concentration of corrosion products in the eutectic regions. Sometimes one finds that all the corrosion products on the surface spall off, giving a surface with clearly metallic appearance. At first sight one might take the metallic lustre as a sign that no corrosion had taken place, but examination under the microscope easily proves that this is not the case, and that the metallic surface has indeed suffered attack.

Thus it is seen that the matrix attack in these alloys assume three different appearances: (1) black regions with very little corrosion, which give no reflections of polarized light under crossed nickels, (2) regions of metallic appearance, and (3) grey regions with aggregates of corrosion products.

By polishing off the outer layer of the samples, it was found that the material in the eutectic had been corroded more deeply than the material in the matrix. Figure 3 shows the same sample as on Figure 2 after some mechanical polishing, and Figure 4 shows the results of removing still more of the surface layer. The width of the corroded zone decreases with increasing depth and the impression is that of a V-notch, although the lower tip of the notch does not appear to be sharp, and is probably as broad as the eutectic region. It is evident that the attacked region at the surface (Figure 3) is much broader than the eutectic region itself (as seen on Figure 1). When comparing with other forms of attack on aluminum alloys, the attack around the eutectic regions is, as will be seen from the micrographs, very even and smooth. One more thing to note about this attack is that the corrosion products which fill up the notches seem to be a very dense material.

Experiments lasting for longer periods of time, up to 834 hours, did not reveal any significant additional features of the corrosion process, except that some small blisters developed. It was not possible to obtain evidence as to the relation between the location and shape of the blisters and the structure of the alloy. Consequently, it is not known if

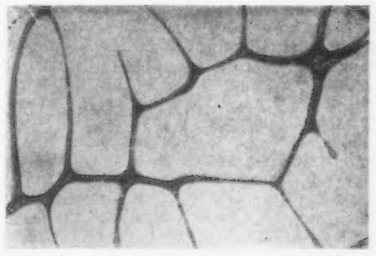

Figure 1—Structure of as-cast Al-0.92 percent Ni alloys. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

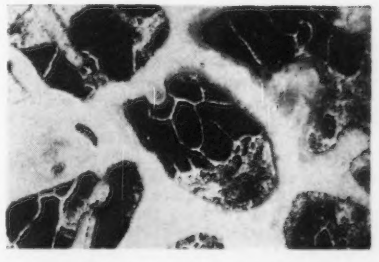

Figure 2—Cast Al-0.92 percent Ni alloy after 18 hours at 300 C in distilled water. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

About the Author

KURT M. CARLSEN is with the Pigments Department, Experimental Station, E. I. duPont de Nemours and Co., Wilmington, Delaware. He was graduated in 1951 from the Technical University of Trondheim, Norway, and spent a year as assistant to Professor A. B. Winterbottom. He did research work in Great Britain at the University of Sheffield under Professor R. W. K. Honeycombe and at the University of Cambridge under Dr. U. R. Evans as a Norwegian Government Research Fellow. Mr. Carlsen also worked for two years as metallurgist with the Dutch-Norwegian Atomic Energy Establishment in Norway.

★ Submitted for publication August 17, 1956.

Figure 3—Same specimen as shown in Figure 2 after some mechanical polishing. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

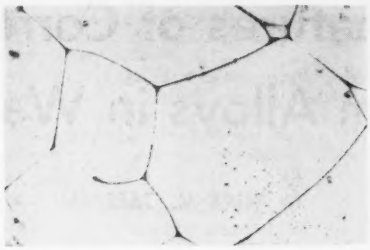

Figure 5—Structure of as-cast Al-0.09 percent Ni alloy. Original magnification, 72X; actual magnification after reduction for engraving purposes, 46X.

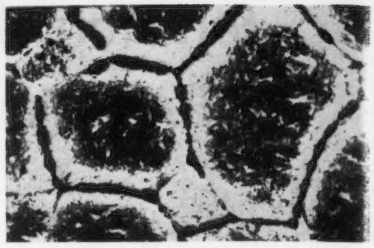

Figure 7—Same specimen as shown in Figure 6 after some mechanical polishing. Original magnification, 72X; actual magnification after reduction for engraving purposes, 46X.

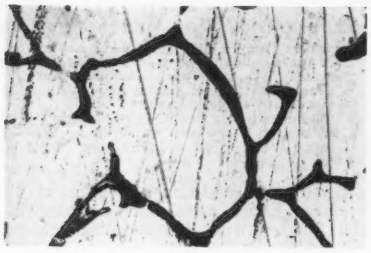

Figure 4—Same specimen as shown in Figure 3 after additional mechanical polishing. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

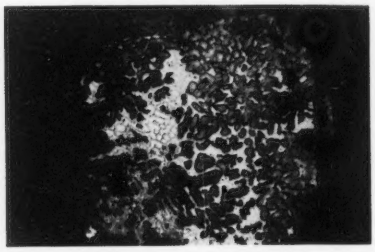

Figure 6—Cast Al-0.09 percent Ni alloy after 1½ hours at 300 C in distilled water. 3X.

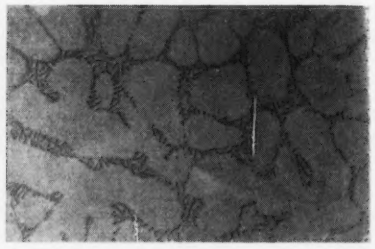

Figure 8—Structure of as-cast Al-0.85 percent Fe alloy. Original magnification, 72X; actual magnification after reduction for engraving purposes, 86X.

these blisters arise for the same reasons as the accelerated attack on Al-0.09 percent Ni alloy described below.

The structure of an Al-Ni alloy containing 0.09 percent Ni is shown in Figure 5, and it is readily seen that the eutectic regions are much less extensive than in the 0.92 percent Ni alloy. The 0.09 percent Ni alloy disintegrates rapidly in water at 300 C, and this disintegration is caused by a very strong attack setting in in the pure aluminum matrix. However, before this accelerated attack sets in, the eutectic regions already have suffered some attack. Figure 6 gives some idea of the development of attack. In the middle of the specimens one can distinguish regions in which eutectic attack is still the important mode of attack. Elsewhere the strong matrix attack has set in; Figure 7 shows the nature of this attack at a higher magnification after removal of the top layer by means of mechanical polishing.

It is interesting to note that this alloy also suffers severe attack at 200 C. In this case, however, the damage is caused primarily by attack spreading out from the eutectic regions, and the matrix attack is not a very important part of the total amount of attack.

An alloy containing 4.5 percent Ni also was used in a few experiments. In the initial stages the appearance of the alloy was very similar to the Al-0.92 percent Ni alloy, with a black layer in the matrix and attack in the eutectic regions. However, this attack did not penetrate by far so deeply into the metal as was the case with the 0.92 percent Ni alloy. After 124 hours, accelerated attack similar to the type met with in the 0.09 percent Ni alloy was observed. The microscopic evidence was not quite conclusive, but there were indications that this accelerated attack started in the matrix.

In another series of experiments, above 310 C, cast Al-1 percent Ni alloys were found to suffer the same kind of accelerated matrix attack which finally led to complete disintegration of the specimens. A rolled Al-Ni alloy containing 1.04 percent Ni, annealed at 450 C for one hour for recrystallization, was tested at 300 C for 834 hours. The microstructural observations indicated strongly that at this stage, attack was concentrated in the eutectic regions, in accordance with the observations on cast alloys. No blisters were observed on rolled alloys.

Al-Fe Alloys

The structure of cast Al-0.85 percent Fe alloy is shown in Figure 8. This material is very similar to the Al-0.92 percent Ni alloy, with the difference that the particles in the eutectic regions appear to be larger and less closely spaced in the iron-containing alloy than in the nickel-containing one.

Also in the corrosion patterns there are some differences. Figure 9 shows that matrix attack is more serious than in the 0.92 percent Ni alloy. It might indeed seem from the micrograph that the matrix is more attacked than the eutectic. However, on polishing off the top layer of the sample, it was found that penetration of corrosion in the eutectic regions is deeper than in the matrix, although the difference is not so pronounced as in the Al-Ni alloys, as seen in Figure 10. This difference between Al-Ni and Al-Fe alloys was confirmed in experiments lasting up to 834 hours. After being exposed for some time, Al-0.85 percent Fe alloy suffered extensive damage due to accelerated attack in which the metal surface was broken up, leaving a mixture of metal and corrosion products.

Black or metallic matrix regions were not observed on Al-Fe alloys.

An Al-14.5 percent Fe alloy with large primary crystals of Fe_2Al_7 also was tested. The impression was that the Fe_2Al_7 crystals were much more resistant to attack than the $NiAl_3$ crystals under the exposure conditions in which the alloys were tested.

Al-Ni-Fe Alloys

An alloy containing 0.5 percent Ni and 0.5 percent Fe was prepared which had the microstructure shown in Figure 11.

In general, samples of this alloy show the three kinds of matrix appearance as did the Al-0.92 percent Ni alloy, but there are important differences in behavior. While matrix attack appears to be less strong than the attack in the second-phase regions, both are definitely of a much smaller magnitude than the attack on the Al-0.92 percent Ni alloy; indeed, it is only with some difficulty that the difference in corrosion rate between second-phase regions and matrix can be discovered. Some small blisters were found, but again it was impossible to get a clear impression of their relation to the structure of the alloy. The microscopic examinations might lead to the inference that the blisters were not filled with corrosion products. If so, presumably they were filled with gas.

Al-Cu Alloys

These alloys were much less resistant to corrosion than the above mentioned alloys. The surface was covered immediately with a layer of corrosion products, and blisters were soon formed. At any rate in the early stages of attack, little or no corrosion products could be seen in the blisters. Figure 12 shows a blister formed on a Al-0.89 percent Cu alloy after 105½ hours' exposure.

Al-Ni-Si Alloys

An alloy was prepared containing 0.5 percent Ni and 0.45 percent Si. The microstructure is shown on Figure 13. It is seen that the structure is different from that of the Al-Ni-Fe alloy. In particular, one notices that the second-phase regions are split up in small bits.

In accordance with the results of Draley and Ruther the corrosion resistance of the alloy was found to be in-

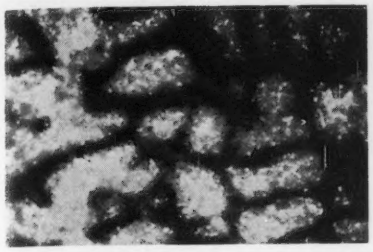

Figure 9—Cast Al-0.85 percent Fe after 16½ hours at 300 C in distilled water. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

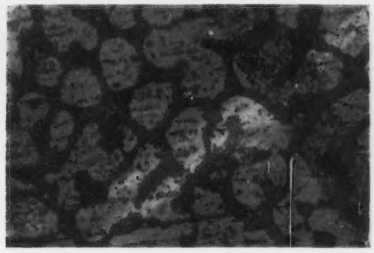

Figure 10—Cast Al-0.85 percent Fe alloy after 17½ hours at 300 C in distilled water. Top layer was removed by mechanical polishing. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

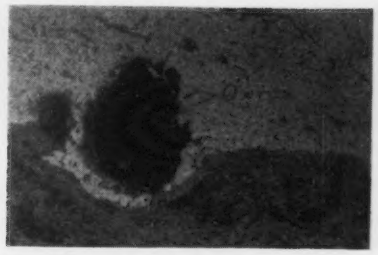

Figure 12—Cast and solution-treated Al-0.89 percent Cu alloy after 105 hours at 300 C in distilled water. Metal is at top and mounting medium is shown in lower portion of photograph. Original magnification, 72X; actual magnification after reduction for engraving purposes, 46X.

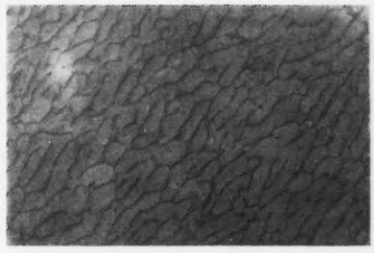

Figure 11—Structure of as-cast Al-0.5 percent Ni-0.5 percent Fe alloy. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

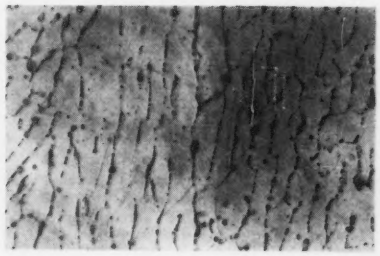

Figure 13—Structure of as-cast Al-0.5 percent Ni-0.45 percent Si. Original magnification, 135X; actual magnification after reduction for engraving purposes, 86X.

erior to that of the Al-Ni-Fe alloy. No structural details of the corrosion process could be revealed clearly.

Discussion

Although the beneficial effect of cathodic alloying elements on the corrosion of aluminum at high temperatures has been reported by several investigators, the way in which these constituents influence the general (non-blistering) attack has not received much attention. This is true also of the question of optimum dispersion of the cathodic phase with regard to corrosion resistance. The only reference to the latter point comes from Draley[2] who stresses the importance of having a finely dispersed precipitate.

It is not clear whether or not Draley's remark applies both to blistering attack and to ordinary general attack. If it does apply to both, one might think that the general corrosion resistance around the precipitates is better than in the pure Al matrix.

The present results show that on cast alloys, at least, the matter is not so simple. Contrary, perhaps, to what would be expected, it is found that the aluminum metal in the eutectic Al-Ni regions corrodes faster than anywhere else. Furthermore, the evidence seems to suggest that the smaller the amount of eutectic, the greater the attack on the eutectic. (This does not necessarily apply to the accelerated attack which sets in the pure Al-matrix.) It might look, therefore, as if the ratio of areas of pure Al-matrix to eutectic regions on the surface to some extent determines the general corrosion rate at least to start with. The higher this ratio, the stronger the attack in the eutectic regions. From this point of view the importance of fine dispersion (i.e., of large surface) of the precipitate becomes easily understandable.

It is generally accepted that the extensive blistering damage on high purity aluminum is cathodically stimulated damage. Since the $NiAl_3$-particles in Al-Ni alloys presumably act as cathodes, it looks as if the protection afforded to some extent is sacrificial protection. Instead of accelerated cathodic attack possibly due to hydrogen the result is a more general cathodic attack.

This latter attack in the author's view definitely is not of the hydrogen type. The whole topology of attack and the smoothness with which it occurs points strongly against the occurrence of hydrogen blisters. More likely, the attack could be stimulated by cathodically produced alkalinity.

Draley[2] seems to suggest that the good corrosion resistance of Al-Ni-Fe alloys is due to the location of a nickel-containing precipitate at grain boundaries and of an iron-containing one within the grains. It is doubted, however, that such an explanation is correct for cast alloys in the initial stages of attack.

First, by comparing the structures in Figures 1 and 8, it is difficult to see any great differences between Al-Ni and Al-Fe in the way in which the precipitate is distributed in the as cast-condition. Furthermore, examination of rolled alloys shows that most nickel-containing precipitate is not located on the grain boundaries. On the other hand, the wide spacing of the lamellae in the eutectics of Al-Fe alloys, and the low percentage of iron (1.8 percent) necessary to make a fully eutectic structure (as compared to 5.7 percent for nickel) give larger amounts of eutectic regions in Al-Fe alloys than in Al-Ni alloys of corresponding composition.

Secondly, an Al-Ni-Fe alloy does not give precipitates corresponding to the sum of precipitates in the two binary alloys, Al-Ni and Al-Fe. For instance, the Al-0.5 percent Ni-0.5 percent Fe alloy precipitates only one secondary phase, namely $FeNiAl_9$. The structure of the Al-0.5 percent Ni-0.5 percent Fe alloy is shown on Figure 11, and it is evident that precipitation of the ternary phase takes place on a much finer scale than for the corresponding binary alloys (Figures 1 and 8). The rate of solidification is identical for the three structures.

It is believed, therefore, that the corrosion behavior of Al-Ni-Fe must be explained in terms of the fine distribution of ternary precipitate in these alloys.

The inferior resistance of Al-Ni-Si alloys is not so easily understood. According to the phase diagram, the ternary alloy should give the same precipitates as the corresponding binary alloys. However, when comparing Figures 1 and 13 (same rate of solidification) it is evident that the presence of Si greatly alters the distribution of nickel-containing precipitate. How this affects the corrosion resistance cannot be decided upon at the present time.

A closer study of Al-Cu alloys might yield valuable information as to the role of dispersion of precipitate.

The poor corrosion resistance of solution-treated and aged Al-Cu alloys show that very fine dispersion of a cathodic precipitate is not in itself a guarantee of low general corrosion resistance, not even for avoiding blistering corrosion. In this connection it would be of interest to compare the effectiveness of Cu as an alloying agent with that of Cu-salts used as aqueous inhibitors.

At present, little can be said about the true mechanisms of corrosion of the Al-matrix in the alloys investigated here.

The structure of the thin black layer is not known; diffraction data reveal only boehmite, but that may not be the actual composition and structure of the layer. Also it is not known if the matrix attack is cathodically or anodically stimulated. If it is cathodically stimulated, it must be so in a way different from the accelerated matrix attack found on the Al-0.09 percent Ni alloy.

Acknowledgment

The author expresses his thanks to T. O. Wullf for his kind assistance in carrying out the experiments.

References

1. J. E. Draley and W. E. Ruther. Proceedings of the Geneva Conference on Peaceful Uses of Atomic Energy, **9**, 391; *Corrosion*, 12, 480t-490t (1956) Oct.
2. J. E. Draley. *Ibid.*, **9**, 447.

DISCUSSION

Comments by J. E. Draley and W. E. Ruther, Argonne National Laboratory, Lemont, Illinois:

Care must be taken to distinguish between the initial stages of reaction (where it is shown most of the attack

is in the eutectic zone) and long continuing corrosion. After long corrosion testing, from a few weeks at 200 C to a year at 350 C, the surface is at least roughly uniformly corroded. That is to say, the attack in the eutectic regions does not remain much faster than that of the matrix. It follows that it cannot be deduced from the evidence provided that the depth of attack (or the general corrosion rate) will be increased by a larger ratio of Al-matrix to eutectic area. Hence this would not seem to be a satisfactory explanation for the desirability of fine-grained material.

We still prefer the explanation based on the size of the matrix phase areas which can be protected by cathodes. There are two types of anodic protection which might be effective:

1. To stop blistering, the entry of hydrogen into the metallic aluminum can be prevented.
2. Cathodically produced OH^- should be kept away from the matrix phase to avoid alkaline attack. A few investigators have shown the latter effect at more ordinary temperatures by external polarization; for example Groot (Hanford report HW-33881, The Galvanic Corrosion of Aluminum, 1955).

Optimum anodic protection should be provided by finely dispersed cathodes on the sample surface.

It is somewhat surprising that Dr. Carlsen found no indication of precipitate within the grains for nickel-iron ternary alloys. As indicated in Reference 1, we found such a precipitate in binaries containing iron, but not in binaries containing nickel. In addition, all ternaries examined have shown such a precipitate. The accompanying photomicrograph (Figure 14) shows the typical as-cast structure of an alloy containing 0.5 percent Ni plus 0.2 percent Fe (remainder high purity aluminum). Magnification is 250X. The addition of another 0.1 percent Fe caused considerable broadening of the grain boundary eutectic, but little change in the appearance of the grains. It should be remarked that the intra grain precipitation in the presence of iron has never been seen by causal inspection. It has been necessary to etch carefully, using "modified Keller's etchant" (HNO_3, HCl, HF, H_2O) for times of the order of 30 seconds.

In addition, our alloys have customarily been chill-cast in small molds, with resultant fine structure, and perhaps with precipitation occurring which is different from that obtained by Dr. Carlsen.

Regarding the effect of silicon, there are two effects which are suggested. When this element is added in small quantities to a binary alloy containing iron or nickel, considerable grain refinement should result if typical casting methods are used. This should be beneficial. In addition, as previously discussed,[1] significant quantities of silicon can be expected to dissolve in the matrix phase at the corrosion test temperature. This should make the matrix more cathodic and more difficult to protect anodically.

There is some evidence to suggest that copper also dissolves in the matrix to an extent which helps to explain its relatively poor effectiveness as an additive for the prevention of intergranular or blistering attack.

Reply by Kurt M. Carlsen:

The author appreciates Dr. Draley's and Dr. Ruther's comments on the duration of experiments. Samples examined by the author after five weeks' exposure still showed stronger attack in the eutectic regions than in the matrix regions. Whether five weeks should be considered an "initial stage" or not, is surely a debatable point, but there is every reason to believe that what takes place during this period determines the subsequent behavior of the sample. On the other hand, it would be very interesting to see microscopic evidence that samples are uniformly corroded after longer periods.

With regard to the two types of anodic protection which are mentioned, the author would like to make the following remarks:

Re (1): There is no evidence that hydrogen gas is in any way responsible for the catastrophic corrosion of aluminum of certain purity levels.

Re (2): The statement that "cathodically produced OH^- should be kept away from the matrix phase—" and that this can be provided "by finely dispersed cathodes on the sample surface" seems reasonable enough, but does not offer a satisfactory explanation in the sense of suggesting a specific mechanism for the phenomenon. Obviously, it would seem that by having finely dispersed cathodes one would concentrate rather than keep away OH^- from the matrix phase, and that corrosion rate would increase rather than decrease. It seems reasonable to believe that some yet unformulated mechanism is operative here, and the idea of sacrificial protection was offered by the author in the hope that it might be a lead in the elucidation of this mechanism.

As for the location of second phases in the alloys, the author has not, as is evidenced by the text, disputed the presence of iron-rich precipitate in the grains. What the author has disputed is the notion that somehow iron-containing alloys are markedly different from nickel-containing alloys in this respect, and that ternary alloys will exhibit features which can be derived as a sum of the features of the binary systems.

In the as-cast condition both of the binary alloys will contain eutectic areas between the primary dendrites and between the grains themselves. Mechanical working followed by recrystallization will distribute the second phase particles more homogeneously throughout the new grains. Because of coarser particles and greater inter-particle spacing in the iron eutectic over the nickel eutectic, the former will tend to appear more homogeneous than the latter. The difference between the two eutectics is brought out clearly in the author's photographs. It should be pointed out that no precipitate formed from the solid state will be found, since the solubility of iron and nickel in aluminum is practically independent of temperature.

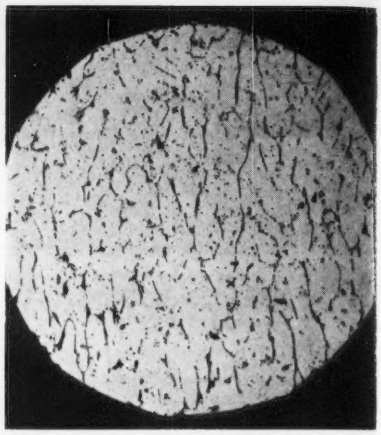

Figure 14—Typical as-cast structure of an alloy containing 0.5 percent Ni plus 0.2 percent Fe (remainder high purity aluminum). Original magnification was 250X; actual magnification after reduction for engraving purposes is 120X. Modified Keller's etch.

The ternary alloys will have a structure which depends on whether the particular alloy lies in a two-phase or a three-phase field. The 0.5 percent Fe-0.5 percent Ni alloy lies in the two-phase region and will solidify in a pseudo-binary manner, similar to the binary alloys mentioned above. Figure 11 shows clearly the ternary eutectic in the ternary alloy, and the similarity with the two binary systems is evident. It follows that the assumption of distribution in a ternary alloy as a sum of the distribution in the corresponding binaries is not correct. Similarly, since the corrosion resistance of this particular ternary alloy is very good, the implied explanation of the improved resistance in aluminum-iron-nickel alloys must be wrong.

Regarding metallographic techniques, the author believes that electropolishing as used in the present investigation is superior to mechanical polishing followed by etching in detecting micro-constituents in aluminum alloys with small amounts of second phases. It looks to the author as if the micrograph shown in Figure 14 is prepared by mechanical polishing and etching. It should be compared with the microstructure shown on Figure 11. The clarity of the latter, as shown for instance in the continuity of the ternary eutectic, speaks for itself.

Any discussions of this article not published above will appear in the June, 1958 issue

San Francisco Technical Committee Program Set

Over 80 Meetings Planned to Cover Principal Industrial Corrosion Problems

Approximately 80 NACE Technical Committees have scheduled meetings during the 1958 Annual Conference in San Francisco. Meetings all day Monday, March 17 will be held in the Fairmont Hotel. Meetings on Tuesday, Wednesday, Thursday and Friday, March 18 through 21, will be held in the Civic Auditorium. Technical committee meeting programs will vary from round table discussions to presentation of formal reports. Following is a brief discussion of what each committee plans to discuss.

Technical Group Committee T-1, Corrosion in Oil and Gas Well Equipment: 9 a.m.-4:30 p.m. Monday, March 17, Fairmont Hotel. T-1 will hear reports from unit committees on various phases of corrosion in oil and gas well equipment. A question-answer discussion period is planned for the afternoon session. T-1 committee members will be asked to submit questions for discussion before the meeting.

Unit Committee T-1B, Condensate Well Corrosion: 1:30-3:30 p.m., Wed., March 19, Civic Auditorium. Unit Committee T-1B work has been concentrated on high pressure well completion and corrosion mitigation procedures during the past year. Task group on the subject will report on the results of two questionnaires designed to bring the committee up to date on high pressure well completion and corrosion mitigation procedures.

Unit Committee T-1C, Sweet Oil Well Corrosion: 3:30-5:00 p.m., Wed., March 19, Civic Auditorium. T-1C will hear reports from groups on water dependent field practices, water dependent corrosion experimental aspects, water independent field practices and water independent corrosion aspects.

Unit Committee T-1D, Sour Oil Well Corrosion: 8-9 a.m. Wednesday, March 19, Civic Auditorium. T-1D will hear reports from task groups on pump corrosion and discuss latest developments on corrosion mitigation in sour oil wells.

Unit Committee T-1F, Metallurgy: 2-5 pm, Thursday, March 20, Civic Auditorium. T-1F will discuss metallurgical problems currently confronting the oil and gas production industry. Task Group 1-1F-1 on Sulfide Stress Corrosion Cracking will report at the meeting immediately following the T-1F meeting. T-1F-2 on Sweet Crude and Condensate Stress Corrosion Cracking will report at the T-1F meeting and will hold a meeting immediately following the T-1F-1 meeting.

Unit Committee T-1H, Oil String Casing Corrosion: 9-10 am, Wednesday, March 19, Civic Auditorium. Latest developments on cathodic protection of oil well casings will be discussed.

Unit Committee T-1J, Oil Field Structural Plastics: 2-4:30 pm, Wednesday, March 19, Civic Auditorium. T-1J will hear reports on performance of plastic tanks and pipe. It is expected discussion on service history from various operating companies will be presented.

Unit Committee T-1K, Inhibitors for Oil and Gas Wells: 10-11 am, Wednesday, March 19, Civic Auditorium. T-1K will hear reports from task groups on sour crude inhibitor evaluation and sweet crude inhibitor evaluation. A method for testing inhibitors for use in oil and gas wells will be discussed.

Unit Committee T-2A, Galvanic Anodes: 9-11 am, Wednesday, March 19, Civic Auditorium. T-2A will discuss latest developments and application of galvanic anodes in cathodic protection installations. It is expected the Task Group on Development of Standard Accelerated Galvanic Anode Test Procedures will report.

Unit Committee T-2B, Anodes for Impressed Currents: 2-5 pm, Thursday, March 20, Civic Auditorium. T-2B will hear reports from Task Groups on Sources of Impressed Current for Anodes, Ground Bed Design Data, Use of High Silicon Cast Iron Anodes and use of Platinum, Palladium, etc. for Anodes.

Unit Committee T-2C, Minimum Current Requirements: 9-11:30 a.m., Monday, March 17, Fairmont Hotel. T-2C will hear reports from task groups on basic criteria, coupons as a criteria for cathodic protection, history of cathodic protection and cathodic protection criteria research. A proposed minimum current requirement for the minimum protection of buried pipe lines will be presented to the committee by a special task group. If approved the report will be submitted to the T-2 Task Group on Minimum Requirements for Protection of Buried Pipe Lines.

Unit Committee T-2D, Standardization of Procedures for Measuring Pipe Coating Leakage Conductance: 9-11:30 am, Tuesday, March 18, Civic Auditorium. T-2D will discuss Task Group reports on Procedures for Measuring pipe coating leakage conductance in short insulated sections of low conductance, at inaccessible locations such as water crossings, with parallel lines and on pipe observed to be in stray current fields.

Unit Committee T-2E, Internal Corrosion of Product Pipe Lines and Tanks: 9-11:30 am, Tuesday, March 18, Civic Auditorium. T-2E will continue to discuss results of the committee sponsored in-line coupon program. Several companies participating in the testing program should have coupons available for inspection and discussion.

Unit Committee T-2H, Asphalt Type Pipe Coatings: 9-11:30 am, Wednesday, March 19, Civic Auditorium. T-2H will discuss committee reports on asphalt type coatings application and procedures, minimum requirments. Task groups on methods to classify environmental conditions, criteria for reporting on and evaluating underground coatings and internal pipe treatment specifications will report.

Unit Committee T-2J, Wrappers for Underground Pipe Line Coatings: 9-11:30 am, Mon-

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Tentative Schedule of Technical Committee Meetings at San Francisco

TECHNICAL COMMITTEE MEETING SCHEDULE—FAIRMONT HOTEL

Monday—March 17	A M	T-1	T-2J	T-2C	T-5A-1 T-5A-3 T-5A-6		T-6G & T-6R	T-3G-1				
	P M	T-1	T-3H	T-4A	T-5A-4 T-5A-5 T-5A	T-6E	T-4F-1 T-4F	T-2H-4				

TECHNICAL COMMITTEE MEETING SCHEDULE—SAN FRANCISCO CIVIC AUDITORIUM

Tuesday—March 18	A M	T-8	T-3F	T-4B-6 T-4B-1 T-4B-2	T-3G	T-9	T-2E	T-3C	T-2D			
	P M	T-4B-3 T-4B-5 T-4B-4	T-4E									
Wednesday—March 19	A M	T-7 and Underground Corrosion Coordinating Round Table	T-1D T-1H T-1K	T-6A	T-5B	T-2K	T-5D	T-2A	T-3D-1 T-3D	T-2H		
	P M	T-8	T-1J	T-6B T-6J	T-1B T-1C	T-3A T-3B						
Thursday—March 20	A M	T-3E	T-6C		T-6F							
	P M	T-2B	T-5E	T-1F, T-1F-1, & T-1F-2	T-3E-1	T-6D	T-7E	T-4D	T-6K			

San Francisco—

(Continued From Page 75)

day, March 17, Fairmont Hotel. T-2J will discuss material specifications that have been prepared by task groups on asbestos felt, glass wrap, glass base, outer wrap and rock shield. If approved by the committee the reports will be submitted to T-2 Task Group on Minimum Requirements for Protection of Underground Pipe Lines.

Unit Committee T-2K, Prefabricated Plastic Film for Pipe Line Coatings: 9-11 am, Wednesday, March 19, Civic Auditorium. T-2K will hear reports from task groups on standards research and development and history and results. A report on minimum required material specifications and application procedures for prefabricated plastic films for pipe line coating will be discussed.

Unit Committee T-3A, Corrosion Inhibitors: 2-3 pm Wednesday, March 19, Civic Auditorium. T-3A will discuss case history data and will hear reports from task groups on use of chemical inhibitors.

Unit Committee T-3B, Corrosion Products: 3-4:30 pm, Wednesday, March 19, Civic Auditorium. T-3B will discuss the committee sponsored bibliographies of corrosion products and future work.

TECHNICAL REPORTS

on

SULFIDE CORROSION

in the

PETROLEUM INDUSTRY

T-5B-2 Effect of Sulfide Scales on Catalytic Reforming and Cracking Units. Part 1—Metallographic Examination of Samples From a Catalytic Reforming Unit. Part 2—Intergranular Corrosion of 18-8 Cr-Ni Steel as a Result of Hydrolysis of Iron Sulfide Scale. A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Pressures and Temperatures in the Petroleum Industry. Per Copy $.50.

T-5B-2 Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From M. W. Kellogg Co., New York by G. Sorell and W. B. Hoyt. Pub. 56-7. Per Copy $1.50.

T-5B-2 High Temperature Hydrogen Sulfide Corrosion in Thermofor Catalytic Reformers—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From Socony Mobil Oil Co., Inc., Res. & Dev. Lab., Paulsboro, N. J. by E. B. Backensto, R. D. Drew, R. W. Manuel and J. W. Sjoberg. Pub. 56-8. Per Copy $1.00.

T-5B-2 Effect of Hot Hydrogen Sulfide Environments on Various Metals—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From Sinclair Res. Lab. Inc., Harvey, Ill. Pub. 57-2. Per Copy $.50.

T-5B-2 High Temperature Sulfide Corrosion in Catalytic Reforming of Light Naphthas—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From Humble Oil & Ref. Co., Baytown, Texas. Pub. 57-3. Per Copy $.50.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

1061 M & M Bldg. Houston 2, Texas

Unit Committee T-3C, Annual Losses Due to Corrosion: 9-11:30 am, Tuesday, March 18, Civic Auditorium. T-3C will hear a final report on the committee sponsored questionnaire on the cost of coatings used as corrosion mitigation material. A preliminary report on results of the questionnaire was presented to the committee at a meeting in Pittsburgh on November 12.

Unit Committee T-3D, Instruments for Measuring Corrosion: 9-11 am, Thursday, March 20, Civic Auditorium. Discussions from the task group on electrical holiday inspection of coatings will be heard and the committee will discuss new instruments and applications of instruments for measuring corrosion.

Unit Committee T-3E, Railroads: 9-11:30 am, Thursday, March 20, Civic Auditorium. T-3E will hear reports from task groups on corrosion of railroad tank cars and corrosion in railroad hopper cars. The task group report on corrosion of railroad tank cars will include data on application and performance of various organic linings. The task group on corrosion in railroad hopper cars will discuss tests of cathodic protection on railroad hopper cars and use of various metals for hopper cars.

Unit Committee T-3F, on Corrosion by High Purity Water: 9-11:30 am, Tuesday, March 18, Civic Auditorium. T-3F will hear reports from task groups on facilities for production of high purity water, inhibitors, corrosion products, general corrosion problems, declassification and bibliographies. The committee is sponsoring a four-paper symposium, Tuesday afternoon, March 20 at the Civic Auditorium. Papers to be presented will be discussed at the committee meeting.

Unit Committee T-3G, Cathodic Protection: 9-11:30 am, Tuesday, March 18, Civic Auditorium. T-3G will hear reports from task groups on cathodic protection of hull bottoms of ships, cathodic protection of heat exchangers and cathodic protection of process equipment. A report by the cathodic protection of hull bottoms of ships task group approved by the committee will be submitted for publication soon. The task group on cathodic protection of heat exchangers has also concluded a formal report expected to be submitted for CORROSION early this year.

Unit Committee T-3H, Tanker Corrosion: 2-4:30 pm, Monday, March 17, Fairmont Hotel. T-3H will hear discussions from operating companies on procedures used to mitigate corrosion in their tankers. New developments on mitigation of tanker corrosion will be discussed.

Unit Committee T-4A, Effects of Electrical Grounding on Corrosion: 2-4:30 pm, Monday, March 17, Fairmont Hotel. T-4A will hear status reports from task groups on recommendations for materials to be used for electrical grounding to reduce corrosion and recommendations on grounding on private premises.

Unit Committee T-4B, Corrosion of Cable Sheaths: 9 am-5 pm, Tuesday, March 18, Civic Auditorium. T-4B meeting will be devoted to reports of and discussion of reports from task groups on lead and other metallic sheaths, cathodic protection, test and survey, protection of pipe type cable, non-metallic sheaths and coatings, stray current electrolysis. Approximately one hour will be devoted to the work of each group. T-4B activities cover all aspects of corrosion of underground cables used by the utility and communications industries.

Unit Committee T-4D, Corrosion by Deicing Salts: 2-4 pm, Thursday, March 20, Civic Auditorium. T-4D will hear reports from task groups on procedures for conducting field tests below ground and coordination of field programs between cities and transportation companies. Increasing use of deicing salts in various areas and their effect on buried cables and pipe will be discussed.

Unit Committee T-4E, Corrosion by Domestic Waters: 2-3 pm, Tuesday, March 18, Civic Auditorium. T-4E will hear a report from the Task Group on Hot Water Tank Corrosion. Committee project on tests of various domestic waters and their corrosive effect on various metallic materials will be discussed.

Unit Committee T-4F, Materials Selection for Corrosion Mitigation in the Utilities Industry: 3-4:30 pm, Monday, March 17, Fairmont Hotel. The T-4F meeting will be immediately preceded by a meeting of the task group on materials selection in the water industry.

Unit Committee T-5A, Chemical Manufacturing Industry: 9 am-5 pm, Monday, March 17, Fairmont Hotel. The T-5A meeting will be devoted to reports and discussion of reports from task groups on sulfuric acid, acetic acid, Chlorine, nitric acid and HF corrosion. Problems to be discussed by the various task groups include those of users of the various chemicals as well as problems of the chemical manufacturing industry. The nitric acid and HF corrosion task groups have preliminary formal reports to be discussed.

Unit Committee T-5B, High Temperature Corrosion: 9-11 am, Wednesday, March 19, Civic Auditorium. T-5B will hear reports from task groups on oil ash corrosion and corrosion by molten salts and metal. Various problems of high temperature corrosion will be discussed.

Unit Committee T-5D, Plastics Material of Construction: 9-11 am, Wednesday, March 19, Civic Auditorium. T-5D will hear reports from task groups on inorganic acids, inorganic alkalies, gases, water and salt solutions, organic chemicals, engineering design, methods of criteria for evaluating plastics in chemical environment. Results of a questionnaire on use and application of plastic materials of construction by the chemical industry will be presented. The questionnairs was distributed by the committee in 1955. Data have been correlated and will be presented to the committee prior to the meeting.

Task group on engineering design will outline the status of work being done on a standard plastic numbering system.

Unit Committee T-6A, Organic Coatings and Linings for Resistance to Chemical Corrosion: 9-11 am, Wednesday, March 19, Civic Auditorium. T-6A will hear a tentative report on Hypalon coatings, a final report on coal tar coatings, a tentative report on phenolic coatings and a tentative report on polyethylene coatings. In addition the committee will hear a discussion on uses and application of fluorcarbon coatings. Task groups on heavy linings, vinyl coatings, vinylidene chloride polymers, rubber and elastomer coatings, silicones, furanes, epoxys, chlorinated rubbers, organic brick covered coatings, rigid vinyls and polyurethanes will be presented. These reports will consist of status reports of work being done by the task group.

Unit Committee T-6D, Industrial Maintenance Painting: 2-4 pm, Thursday, March 20, Civic Auditorium. T-6D will hear a final report on results of a questionnaire on Plant Painting Programs. A preliminary report on economics of maintenance painting will be discussed. This report is the result of a questionnaire distributed by the economics of maintenance painting task group to over 700 companies in the United States. The task group on the standardization of scope of painting specifications will present its tentative painting specification report.

Unit Committee T-6F, Protective Interior Linings Application and Methods: 9-11:30 am, Thursday, March 20, Civic Auditorium. T-6F will hear reports from task groups on curing, surface preparation, inspection, safety and application.

Unit Committee T-6G, Surface Preparation for Organic Coatings and Unit Committee T-6R, Protective Coatings Research will hold a joint meeting from 9 to 11:30 am, Monday, March 17 in the Fairmont Hotel. These committees will discuss developments in surface preparation techniques and areas of protective coating technology not now covered by the T-6 committees.

Group Committee T-7, Corrosion Coordinating: 9-11 am, Wednesday, March 19, Civic Auditorium. T-7 will hold a round table discussion. The philosophy of coordinating committee work will be discussed and members from various electrolysis and coordinating committees throughout the United States are expected to attend to discuss their problems with the national NACE Committee.

Group Committee T-8, Refining Industry Corrosion will hold two meetings. The first will be from 9 to 11:30 am, Tuesday, March 18 at the Civic Auditorium. The second will be Wednesday, March 19 from 2 to 4:30 pm at the Civic Auditorium. Agenda for the T-8 meeting has not been prepared but it is expected the committee will discuss the napthenic acid corrosion, acid cleaning, high temperature and pressure sulfide corrosion and other problems facing the refining industry today.

T-8 also will sponsor a round table discussion as the second part of the refinery industry symposium. This will be held on the morning of Wednesday, March 19 at the Civic Auditorium.

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Sweet Crude Oil Failures May Be Stress Cracking

Sulfide Cracking Is Unlikely, Survey Results Indicate

At the October, 1957 meeting in Oklahoma City of T-1F-2 on Sweet Crude and Sweet Condensate Stress Corrosion Cracking results of some replies to a questionnaire were presented. Only five replies were canvassed and of these three reported failures. In all 21 failures in sweet crude systems were reported.

Materials involved in the failures were 9 percent chrome- ½ percent molybdenum tubing, 9 percent nickel tubing and 5 percent nickel tubing. Longitudinal splits and circumferential cracks were reported as were both inter and transgranular types of cracking. In four cases failures definitely were reported as stress corrosion cracking and in one case the cause was uncertain. In all cases laboratory attempts to reproduce failures were unsuccessful.

It was requested that the task group circulate additional questionnaires so further data could be accumulated.

Low Hardness Tubing Fails

One member of the task group reported that his company had found stress corrosion cracking failures in 9 percent nickel tubing with hardnesses as low as Rockwell "C" 23 in sweet condensate wells. He said one failure occurred in N-80 tubular goods with a Rockwell "C" hardness of 22. This failure was initiated at straightener roll marks outside of tubing. He reported stress corrosion cracking failure in a sweet condensate well of 4340 steel with physical properties of 105,000 to 125,000 yield strength which had been pressure tested to 10,000 psi prior to installation.

After failure the entire string was pressure tested to 5,000 psi when 87 joints tested, 23 failed. Examination showed many of these joints contained severe, highly branched stress corrosion cracks. The high branching type of crack was unlike those found in sulfide corrosion cracking. The wells had a partial pressure of carbon dioxide of 12,000 psi. Cracks occurred as deep as 8,000 feet, where the temperature is as high as 200 F.

It was questioned whether high strength materials could be used in these applications at all. This member asked if other companies were having this type of failure and if such failures were not being reported as stress corrosion cracking failures because it is not realized that this type of failure is occurring. One member of the committee thought that if high pH mud was present it was possible that the failures might be from caustic embrittlement. Although high pH mud was used in drilling the well, because of mixing and other things that might have occurred, the condition of the mud during the time the well was in operation was uncertain. It had been considered that failures might be due to caustic embrittlement, but the 4340 steel cracked on the inside rather than on the outside, which would indicate that the well fluid and not the mud promoted cracking. The pH of these well fluids with the partial pressure of CO_2 present must be less than 4.

Low Hydrogen Factor Important

Another member asked whether it was known definitely that these wells contained small quantities of H_2S. No evidence of H_2S was found in copper strip tests by plants handling the gas. It was pointed out also that the nature of the cracking differed from that encountered in sulfide corrosion cracking in that it was substantially more branched.

One member said that if the time to failure was delayed considerably by low rate of hydrogen pick up by the steel because of the very low hydrogen available, branch cracks could be expected to an extent much greater than in areas where the time to failure is rapid because of high availability of hydrogen resulting from the presence of hydrogen sulfide. Further discussion indicated the

(Continued on Page 78)

San Francisco—

(Continued From Page 76)

Group Committee T-9, Marine Biological Deterioration: 9-11:30 am, Tuesday, March 18, Civic Auditorium. T-9 will devote much of the meeting to formal organization of the committee's Unit Committee on Biological Aspects and Preservations and Their Appraisal. This is the first formal meeting of Group Committee T-9 at a NACE Conference. Many of the members will be prepared to present discussions on various aspects of marine biological deterioration.

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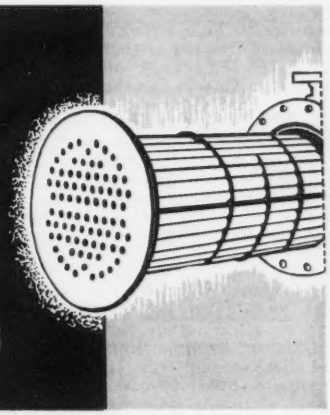

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Oil Well Metallurgical Programs Discussed by T-1F

Definition of Tubing Normalizing Said to Require Revision

Materials for high temperature production, full-length normalizing of upset tubing, nickel-molybdenum and carbon-manganese steel sucker rods in Western Kansas sour oil service, alternate rod mixed-string testing effectiveness, case hardened sucker rod couplings, normalized intermediate alloy sucker rods in sweet corrosive service, corrosion fatigue testing, French steel for sulfide stress corrosion, new API and proposed high strength steels, corrosion stress cracking observation and welding accessories to high strength casings were subjects discussed at the Unit Committee T-1F Metallurgy meeting held in Oklahoma City in October.

In thermal recovery process production it has been found that the flame front had reached producing wells in some instances. Extremely high temperatures are encountered by the down-the-hole equipment in such wells. Selection of materials for this service was considered to be of concern to the committee for future work. One member, in discussing his company's approach to the problem, said its work had been in two directions. One involved consideration of materials which would be satisfactory in high temperature service and the second was consideration of thermal recovery process methods which would eliminate or reduce severity of the problem. The company was still having difficulty with the problem however, and considers it unsolved.

Serious Problem Indicated

It was indicated the problem could well assume serious proportions as thermal recovery production becomes more common. Another member pointed out that high temperature corrosion problems associated with thermal recovery was a result of the process not behaving according to expectations. Engineers concerned with exploring this method of recovery are continually trying to eliminate unpredicted behaviour.

The committee decided that the problem must be more clearly defined before any action can be taken by the T-1F committee and that this definition must come from companies familiar with the process. It was agreed the item would be retained on the T-1F agenda for future consideration.

Better Definition Needed

In discussing full-length normalizing of upset tubing, desirability of full-length normalized tubular goods from a "ringworm" corrosion standpoint was generally accepted. It was also pointed out that some question has been raised as to the clarity of the API specifications concerning the heat treatment of API tubular goods.

According to one member of the committee the API Standard uses the phraseology "fully heat treated." Engineers in the oil production industry have for some time interpreted this to mean fully normalized after the upset operation. However, a report from a member of T-1 indicated some of pipe mills interpret "fully heat treated" to mean fully heat treated prior to the upsetting operation. These mills usually normalize only the ends of tubes after up-setting. It was pointed out that it had been agreed by API that the phrase in the specifications should be changed to read "normalized after up-setting on all N-80 tubular goods and normalize after up-setting J-55 tubing, if such treatment is requested by the customer." N-80 casing is a non-API product therefore full-length normalization of N-80 casing must be specified by the purchaser.

"Ringworm" Protection Questioned

One member of the committee questioned the validity of the conclusion "ringworm" corrosion of J-55 tubing could be prevented by full-length normalizing after upsetting. He said metallographic studies of J-55 showed corrosion to be at the juncture of the large grain to the small grain material of the heat transition zone. Metallographic studies of the effects of a regular normalizing treatment on material that had been treated at forging temperatures indicated that homogenity of the microstructure was not obtained by one regular normalizing treatment. He had found that an analysis of the latter presented in a statistical study of corrosion problems prepared by a member of T-1 could be arranged in such a fashion as to indicate that the "ringworm" problem was not appreciably effected by the normalizing treatment.

It was pointed out also that in the report the breakdown of the strings into non-normalized and normalized is questionable because the author was not sure how the strings actually were heat treated. For this reason, the conclusion that "ringworm" corrosion of J-55 tubing after full-length normalization was not entirely validated by the report.

The Natural Gasoline Association of America report published on the subject several years ago did not show 100 percent solution of the "ringworm" problem, but did show that its severity was reduced sufficiently to make full-length heat treatment economically desirable. It was pointed out also that in recent years inhibition has masked "ringworm" corrosion to such an extent that it is difficult to tell what the severity would be if no inhibitors were used.

Preferential Corrosion Suspected

One member of the committee said his company has experienced severe external pit corrosion of collars on tubing strings where upset line pipe was being used. The collars were of J-55 material, whereas the pipe was of a line pipe material. In one string 35 collars were replaced for each joint of pipe replaced. He raised the question as to whether there might be some preferential corrosion of the higher carbon J-55 material in the collar relative to that in the pipe. There had been no tendency for "ringworm" corrosion in the pipe even though the upset pipe had not been fully heat treated.

One member of the committee thought the preferential corrosion of collars is usually related to turbulence at the collars and the poor spheroidic microstructure characteristic of collar materials. Another member indicated that the problem might be galvanic corrosion between the high and low carbon materials involved in the collar and the tubing. He said that the fluid contained small amounts of hydrogen sulfide and that the corrosion on the collars was characterized by large pits. Most corrosion on the line pipe occured in centers rather than at ends. There was no evidence of attack in the upset regions.

External Collar Corrosion Common

Another member pointed out that external corrosion of collars was not a new experience and that external corrosion of collars in the anulus space occurred in almost every case where the annulus was a vapor space. He cited one instance where all collars and strings had been replaced, but not one joint of pipe. He did not know the cause of the problem but said that, in general, where

(Continued on Page 79)

Sweet Crude Oil—

(Continued From Page 77)

nature of the cracking was so striking it appeared certain a different problem than sulfide corrosion cracking was involved.

It was also noted that when a problem presented was new in appearance and origin it should be considered as a new problem until it had been demonstrated definitely that it is a ramification of an old problem. One member pointed out that the role of H_2S in stress corrosion cracking was to generate hydrogen which, upon diffusion into metal, created a degree of embrittlement. To his knowledge no hydrogen absorption occurs in CO_2 environment. He pointed out that other materials might well be bringing about the hydrogen absorption in the CO_2 environment.

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Oil Well Metallurgy—

(Continued From Page 78)

this problem is encountered no fluid is involved. This fact would tend to exclude the possibility of galvanic action. He felt that the problem possibly relates to condensation of fluid in the annulus and an accumulation of condensate around the collars.

The member who had presented the problem said that he was not using the term "galvanic action" in the sense of bimetallic galvanic action but more with reference to concentration cell effect. He pointed out that the string was in a slant hole and that the collars did go out on one side of the pipe only. He thought this indicated that a film of water was running down on the string on one side, but for some reason all the attack had occurred on the collars and very little on the tubing proper. He also re-emphasized the fact that even though the tubing was not full-length normalized after upsetting, there was no evidence of "ringworm" corrosion although it was obvious that the environment was corrosive from the condition of the collars. He also pointed out that tubing in the string was anchored so that there would be very little movement.

The problem of full-length normalization of upset tubing was retained on the T-1F agenda for future meetings and the problem of external corrosion of collars in the vapor space of tubing strings is to be put on a future T-1F agenda.

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CORROSION's Technical Section published 190 more pages in 1957 than in 1956.

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North Central Region NACE meets in Cincinnati in 1958.

REPORT ON PLASTIC MATERIALS COMPILED

At the meeting of Technical Unit Committee T-5D on Plastic Materials of Construction on March 11, 1957, S. W. McIlrath, chairman, announced that data from the committee's field questionnaire has been compiled. A report will be prepared for presentation at the T-5D meeting in San Francisco. A T-5D suppliers' questionnaire has been prepared and will be distributed in April of 1958.

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Chicago Underground Corrosion Group Elects New Officers

The Chicago Area Committee on Underground Corrosion Committee of NACE Technical Unit Committee T-7B, North Central Region Corrosion Coordinating Committee has re-elected L. E. Nichols, Northern Illinois Gas Company as chairman and J. Svetlik, Northern Indiana Public Service Company, vice-chairman for 1958. They have served as chairman and vice-chairman since the committee's inception.

M. D. Fletcher, Illinois Bell Telephone Co. was appointed secretary for the same period.

At the committee's meeting in October, the chairman appointed a map committee consisting of J. Svetlik, Northern Indiana Public Service Company, V. M. Kalhauge, Standard Oil Company of Indiana, M. D. Fletcher, Illinois Bell Telephone Company and L. E. Nichols, Northern Illinois Gas Company. A publicity and publications committee was appointed after a discussion of methods to enable the committee to secure advance routing of new pipe lines to be constructed in their area. It was suggested that this committee publish a directory of the personnel of member companies who could be contacted by parties constructing new pipe lines or subsurface structures in the area. Personnel to be listed could include those interested in protecting the existing structures from physical damage as well as corrosion.

It was also suggested that forms be prepared by the committee for protective installations notification to members and notification of planned routine and joint testing among the members. Another suggestion was the development of a news letter for publication between meetings with each company as a contributor.

Companies constructing new pipe lines in the Chicago area can save time and money by availing themselves of the information compiled by this committee relative to location of existing lines and present corrosion control systems.

The committee officers requested that each member working at the job of preventing corrosion furnish the committee with the name of the planning engineer of his company so that the information, records and aims of the committee can be given full consideration in advance planning of new pipe lines.

The map committee requested that companies which have an original set of committee maps correct them now to show all new lines, bonds and rectifier installations. This information is necessary in order for the map committee to correct the original tracings and print new copies of the map.

Next meeting will be held January 21, at 208 West Washington Street, Room 1604, Chicago, Illinois, at 1:30 p.m.

CERTIFICATES of MEMBERSHIP in NACE

Certificates of membership in the National Association of Corrosion Engineers will be issued on request at $2 each, remittance in advance. The certificates, which measure 5½ x 8½ inches, are signed by the president and executive secretary of the association.

CERTIFICATES for PAST CHAIRMEN of REGIONS and SECTIONS

Certificates measuring 9 x 12 inches in size, prepared from an engraved plate, are available for issuance to regional and sectional chairmen. They will be supplied on request of the region or section at $7.50 each to be paid by the region or section, the cost to be classified as a non-reimbursable expenditure.

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Louisville Committee Affiliates With T-7C

Gates

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Zell

The Louisville (Ky). Joint Electrolysis Committee has affiliated with NACE Technical Group Committee T-7 Corrosion Coordinating Committee under Unit Committee T-7C Southeast Region Corrosion Coordinating Committee.

The Louisville Committee has been functioning continuously since it was formed in 1924. Present officers are: Stuart H. Gates, Southern Bell Tel. & Tel. Co., Louisville, Kentucky, chairman; John L. Gray, Louisville Gas & Electric Co., Louisville, Kentucky, vice-chairman; Walter Zell, Louisville Gas & Electric Co., Louisville, Kentucky, secretary.

There now are 8 member companies represented on the committee.

Luncheon meetings are held on third Fridays monthly with visits from interested persons welcomed.

San Francisco Area Joint Underground Committee Affiliates

The Joint Committee for the protection of Underground Structures in the East Bay Cities has affiliated with NACE Technical Group Committee T-7 Corrosion Coordinating Committee under Unit Committee T-7E Western Region Corrosion Coordinating Committee.

The Joint Committee for the Protection of Underground Structures in the East Bay Cities officers are: H. C. Snead, San Francisco-Oakland Bay Bridge Toll Plaze, Oakland, Cal., chairman; W. G. Collins, Pacific Telephone & Telegraph Co., San Francisco, vice-chairman; C. K. Notley, Key System Transit Line, Oakland, Cal., secretary-treasurer.

Ray Ditto Is Elected by San Diego County Group

The San Diego County Underground Corrosion Committee of NACE Technical Unit Committee T-7E Western Region Corrosion Coordinating Committee has elected new officers for 1958 as follows: Ray Ditto, California Water & Telephone Co., Chula Vista, Cal., chairman; R. E. Hall, Union Oil Company, Brea, Cal., vice-chairman; F. O. Waters, City of San Diego Water Department, San Diego, Cal., secretary.

Phenolic Coatings Report Circulated

A report on phenolic coatings has been prepared by Task Group T-6A-4 and circulated to Unit Committee T-6A members for comment, criticisms and suggestions. The report deals with two basic types of coatings made from phenolic resins combined with other resins, plasticizers, etc. The two groups are: Straight or baked type phenolic coatings and modified phenolic coatings, either baked or catalytically cured.

Included in the report is a summary of properties, forms available, effect of compounding, testing and selecting, uses, surface preparation and application techniques, failures, inspection, properties of the applied coatings and lining, continuous exposure, toxicity and odor and a chart showing chemical resistance of phenolic coatings in various chemical environments at ambient temperatures.

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The 1959 15th Annual NACE Conference will be held in Chicago.

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Have You Noted The Technical Topics Section?

In every issue of CORROSION a "Technical Topics" section is published. It includes timely technical information likely to interest you. Turn to Page 119 this issue.

NACE NEWS

FORMATION OF AN NACE SECTION in Havana, Cuba was discussed at this meeting in Havana. Seated at the table, left to right, Dr. Rudolph Vonkline, Gutierrez Mendive, H. B. Sasman, Sasman Engineering Co., Miami, Fla. and E. T. Rieser.

Eastern Wisconsin Section Elects Witort, Walczyk and Haase

Eastern Wisconsin Section elected officers for 1958 at its December 4 meeting. New officers are: Edwin A. Witort, Steel Sales Co. of Wisconsin, chairman; David D. Walczyk, A. O. Smith Corp., vice-chairman; Harold F. Haase, corrosion consultant, secretary-treasurer.

Directors elected were C. A. Peter, Western Hardware & Specialty Mfg. Co., for a one-year term; and D. H. Westerman, Miller Brewing Co., and T. E. Quick, Ladish Co., for three-year terms. As part of the business meeting, members heard a short report of section activities.

The meeting was preceded by a cocktail hour and a buffet dinner where 30 members and their wives participated in a brief social period. As part of the evening's program, a Bell Telephone Laboratory color movie, "Our Mr. Sun," was presented. The importance of the sun to human welfare was shown.

At the November 13 meeting, section members heard J. R. Schley, manager, Technical Service Division, Haynes Stellite Co., speak on "Alloys for Severe Corrosion Service." Mr. Schley traced the origin and history of the nickel base alloys and cited a number of examples where they had been used with good economic results. He pointed out that the most logical use for these high alloy materials is in services where the life of other less expensive materials is relatively short, or where contamination from the products of corrosion is an important factor. He further pointed out that these alloys are no miracle materials, but that they had to be applied with judgment and a knowledge of corrosion fundamentals to get the best out of them.

After an interesting discussion period, 15 members and 12 guests adjourned for refreshments. November and December meetings were held at the Engineers' Society Building, 3112 W. Highland Blvd., Milwaukee.

Hungarian Subscriber Sends Good News!

The following reply to an expiration notice was received from a CORROSION subscriber in Hungary:

"Thank you for calling our attention to the subscription of 'Corrosion Magazine'. Hereby I beg to inform you that we have no intention to make a subscription for the year 1958 because of having already solved the problem of corrosion."

San Joaquin Section to Plan '59 Region Meeting

San Joaquin Valley Section will have a principal role in the planning and conduct of the Western Regional Conference to be held in Bakersfield during November 1959. The matter was discussed at the November 20 meeting.

Another project, a three-day field trip of guided tours to Valley field installations in September 1958 also was considered.

NACE Vice-President L. L. Whiteneck, Plicoflex, Inc., Los Angeles spoke on "NACE Looks Ahead." The charter establishing the San Joaquin Valley Section was presented formally by Western Region Chairman and Vice-Chairman, L. E. Magoffin, California Water and Telephone Co. and E. H. Tandy, Standard Oil Co. of California, respectively.

The meeting was held at Maisson Jausaud Restaurant, Bakersfield, with 20 members and 15 guests present.

Inauguration of recently elected San Joaquin Valley Section officers was planned for January, 1958. New officers are Robert L. Davis, Superior Oil Co., chairman; Herbert E. Rose, Superior Oil Co., vice-chairman; and Robert J. Kreps, Standard Pipe Line Co. of Cal., secretary-treasurer. R. I. Stark, Pacific Gas and Electric Co. is past chairman.

Environment Treating Subject for Chicago January Meeting

"Prevention of Corrosion Through Treatment of the Environment," is the subject of a scheduled presentation by David Boies, National Aluminate Co. before a January 21 meeting of Chicago Section.

Fifty-one members and guests were present November 19 to hear Marshall E. Parker, consultant, speak on "Field Evaluation of Pipe Line Coatings." According to Mr. Parker, two basic methods exist for evaluation of coating on a section of pipe line: 1) Measurement of the "conductance to remote earth" and: 2) Determination of the current requirements for cathodic protection. Neither method is, in itself, valid for predicting the future performance of a coating. Neither method yields results dependent on coating alone. Factors such as soil resistivity, line diameter, depth of burial, proximity to other structures and stray currents enter into the value obtained.

Conductance to remote earth is measured by determining for a section of line, under temporary partial cathodic protection, the following two quantities: 1) The total amount of cathodic protection current flowing to the line section, and: 2) The average shift in potential with respect to remote earth, associated with the current flow. In many instances, the determination of the true average potential is the difficult part of the task.

Current required for cathodic protection is determined in a similar manner. Temporary, partial protection is applied, and the results extrapolated to complete protection, Mr. Parker concluded.

Case histories were discussed and pitfalls were brought out in the discussion. He said performance of coatings under cathodic protection is becoming increasingly important and coating manufacturers are working with pipe line engineers to develop the needed coatings.

A lively question and answer period followed his talk.

Harold C. Boone, section vice-chairman introduced Mr. Parker.

Shreveport Section's Short Course Set Feb. 13-14

Shreveport Section's 1958 short course will be held Feb. 13-14 at Centenary College, Shreveport, La. M. J. Olive, General Engineering Dept., Arkansas Fuel Oil Corp., Shreveport, La., is chairman.

At the regular monthly meeting, held Dec. 3 at the Caddo Hotel, 35 members and guests heard O. W. Wade, Transcontinental Gas Pipe Line Co., Houston, speak on "Internal Coatings of Large Diameter Gas Pipe Lines."

Airplane Check on Smog

Twice a day flights by Los Angeles Air Pollution District airplanes to inspect the county for violations of smog laws and photograph violations for subsequent court action began August 5.

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Corrosion Sales Engineer, Pipeline Materials. Growing organization has established Northeastern territory to fill. Married applicant, residing in Northern New Jersey or New York Metropolitan area, given preference. Travel. Salary commensurate with ability. CORROSION, Box 58-1.

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Whiteneck, Godard, Stegner Elected Officers; Gribble, McFarland, Wasson Named to Board

A tally of votes cast on 2435 mail ballots received at Central Office for officers and directors of the National Association of Corrosion Engineers shows the following to have been elected:

President, L. L. Whiteneck, Plicoflex, Inc., Los Angeles, Cal.; Vice-president, H. P. Godard, Aluminium Laboratories, Ltd., Kingston, Ont.; Treasurer, A. L. Stegner, Tennessee Gas Transmission Company, Houston. Directors have been named as follows: C. G. Gribble, Jr., Metal Goods Corp., Houston; R. McFarland, Jr., Hills-McCanna Co., Chicago and L. C. Wasson, A. O. Smith Corp., Milwaukee.

Officers' and directors' terms begin on the last day of the 14th Annual Conference at San Francisco. Officers' terms are one year while directors serve for three.

Long Active in Association

President-elect Whiteneck has long been active in NACE affairs, especially in Western Region, where he has served as regional officer. He currently is vice-president of the association. His corrosion experience includes work with the Long Beach, Cal. Harbor Department and water departments. He presently is vice-president and director of materials research for Plicoflex, Inc.

Hugh P. Godard, head of the chemical division, Aluminium Laboratories, Ltd., Kingston, Ont. has been active for the past 12 years in work connected with the corrosion behavior of aluminum. He is author of many technical papers on aluminum corrosion, is active in NACE affairs as a director of the association and as a member of the abstract and of technical committees. He has been chairman of the Inter Society Corrosion Committee and a member of the Canadian Associate Committee on Corrosion Research and Prevention since its formation in 1947, among other organizations in Canada.

A. L. Stegner, who directs the corrosion control program for Tennessee Gas Transmission Company, Houston, has been active in NACE affairs as a director and otherwise before being elected treasurer for the first time in 1957-58. He is a member of other organizations in the petroleum field concerned with corrosion control.

C. G. Gribble, Jr., joined Metal Goods Corp., Houston after he returned from military service in 1946. He is now Houston district manager of his company, has held offices in Houston Section and has been active in management of arrangements for NACE national conferences. He will represent corporate members on the board.

R. McFarland, Jr., who has spent some 20 years in the design and application of corrosion resisting valves to chemical equipment, is secretary and technical director of Hills-McCanna Company. He has been chairman of Technical Committee T-6A and is co-chairman of the San Francisco Conference technical program committee. He is a member of and active in several other organizations concerned with corrosion control matters. He represents active members on the board of directors.

L. C. Wasson, who worked for 11 years for Milwaukee Gas Specialty in research, development and design of controls for the gas industry, has been with his present employer, A. O. Smith Corp., Milwaukee successively in its electrical research laboratory and as director of its electrochemical laboratory where his work has been on cathodic protection and related phenomena. He represents corporate members on the board.

Positions Wanted

Sales Engineer. Graduate engineer, 12 years' experience—sales, management, operations and maintenance—in industrial and municipal water and waste treatment and corrosion desires sales, marketing and/or public relations position. CORROSION, Box 57-25.

Two Technical Talks Are Given at Los Angeles

Controlled corrosion as a means of removing unwanted metal, and the use of metallized coatings were the subjects discussed by two speakers at the November 20 meeting of Los Angeles Section.

Hugh H. Muller, Chem-Mill Division, Turco Products described the techniques of the chemical-milling of metals using controlled corrosion processes. This method of removing unwanted metal is finding wide use in the aircraft and missile industry, he said.

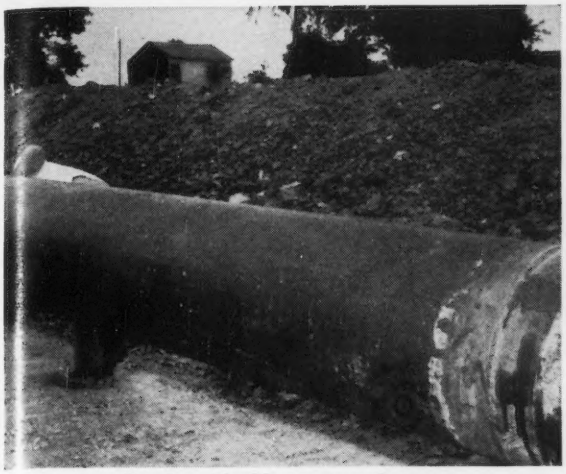
Walter B. Meyer, president of the Metco System Contractors Association, described the techniques of flame spray application of metallic coatings. The use of metallized coatings in combination with organic coating materials was also covered.

Each speaker had a 30-minute presentation period. A question and answer session followed the presentations. About 60 members and guests were present.

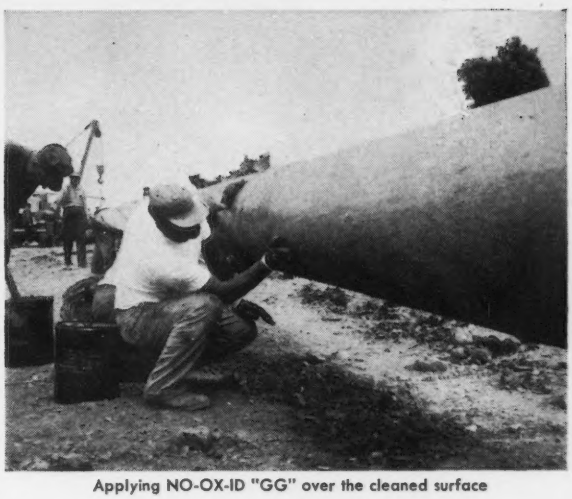
H. H. Uhlig to Talk

H. H. Uhlig, Massachusetts Institute of Technology, Cambridge is scheduled to be featured technical speaker at a February 4 meeting of Montreal Section. Jason J. Waller, Canadair, Ltd., Montreal will speak at the March 4 meeting.

Both meetings are scheduled to be held at the Queens Hotel, Montreal.



Pipe, buried for 25 years, is good as new



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Next, No. 4 NO-OX-IDized wrapper for protection against abrasion

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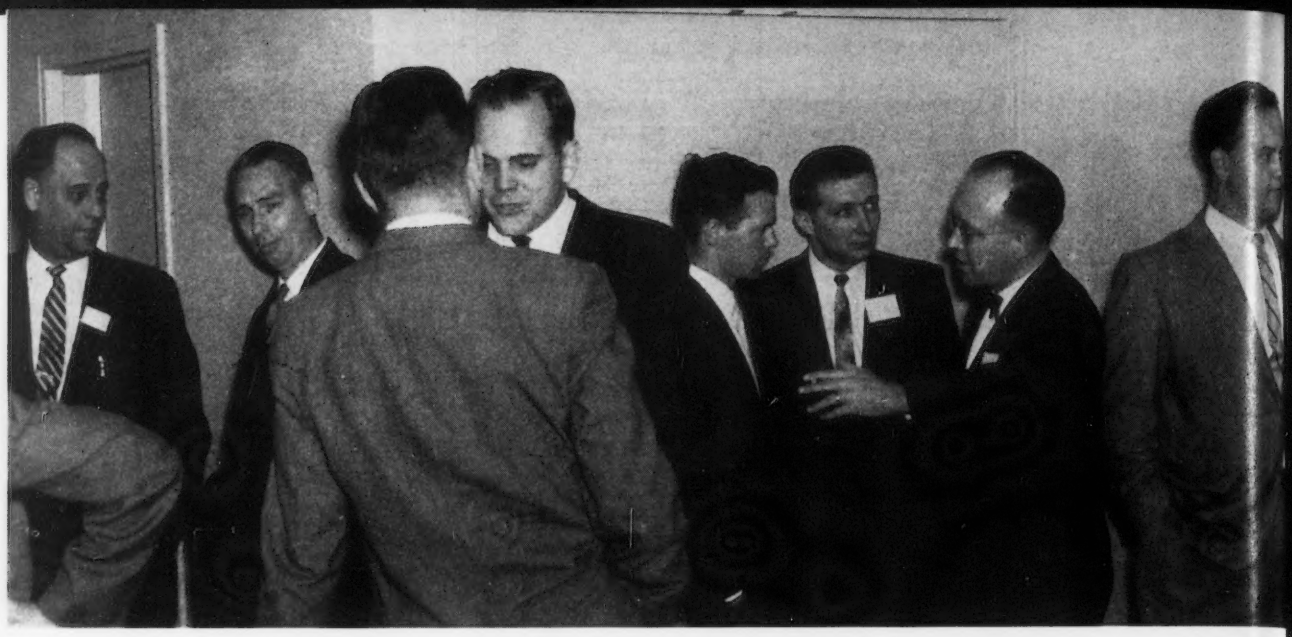

SOME OF THE approximately 30 registrants attending the November 19, 1957 Fall Meeting of Toronto Section are shown. Left to right: Charles Smith, Corrosion Service, Ltd.; Bert Barraclough, Triangle Conduit and Cable (Canada) Ltd.; Lance Heverly, Trans-Canada Pipe Lines, Ltd.; John Trim, Corrosion Service Ltd.; Robert Rhodes, Northern Ontario Natural Gas; George Stone, Glidden Co.; Bob Lockwood, Trans-Canada Pipe Lines, Ltd. and John Balmer, Consumers Gas Co. The meeting heard J. B. Hambley, Alchem Ltd., speak on "Chemical Treatment for Corrosion Control in Industrial Cooling Systems." He described the causes and effects of corrosion and the importance of scaling and microbiological growth, methods of treatment for trouble free operation and modern methods of corrosion measurement. H. A. Webster, Corrosion Service Ltd., presented the Toronto Section's annual award to J. Hazelton, director of the gas technology course at the Ryerson Institute of Technology, Toronto. Prizes will be awarded yearly for the first and second best papers written on corrosion by students of the course.

Illinois Biennial Short Course Held

The Biennial Short Course on Cathodic Protection of University of Illinois was held December 9-13 with the cooperation of NACE.

The program for the short course included sessions relating to elementary and advanced corrosion studies, corrosion mitigation practices, basic electrochemistry, instruments, cathodic protection practices, economics, lead cable sheaths, equipment and reviews of research status.

Participating in the program as lecturers were H. R. Aschan, Good-All Electrical Manufacturing Co., Ogallala, Neb.; F. B. Burns, Kerr-McGee Oil Industries, Inc., Wynnewood, Okla.; B. Douglas, Dow Chemical Co., Midland, Mich.; W. F. Fair, Jr., Koppers Co., Verona, Pa.; Norman Hackerman, University of Texas, Austin; M. Hendrickson, Minnesota Mining & Mfg. Co., St Paul, Minn.;

V. E. Lowden, Illinois Bell Telephone Co., Chicago; W. A. Luce, The Duriron Co., Dayton, Ohio; M. G. Markle, Northern Illinois Gas Co., Bellwood; J. W. Miller, National Carbon Company, Cleveland; L. E. Nichols, Northern Illinois Gas Co.; M. E. Parker, consultant, Houston; V. J. Payton, Commonwealth Edison Co., Chicago; W. J. Schwerdtfeger, National Bureau of Standards, Washington; L. P. Sudrabin, Electro Rust-Proofing Corp., Belleville, N. J.

Faculty members participating were: L. B. Archer, professor of electrical engineering; W. H. Bruckner, research professor of electrical engineering; H. N. Hayward, professor of electrical engineering; O. G. Jansson, research assistant in electrical engineering; E. C. Jordan, head, department of electrical engineering; T. E. Larson, head, Chemistry Section, State Water Survey, Urbana, Ill.; W. W. Litchenberger, research associate control systems laboratory; M. Metzger, research associate professor of metallurgical engineering; R. K. Newton, supervisor, engineering extension, Division of University Extension.

East Texas Section Elects B. G. Hustead Chairman

New officers for East Texas Section, elected November 26 are B. G. Hustead, East Texas Salt Water Disposal Co., chairman; Gene E. Smith, Lone Star Steel Co., vice-chairman; H. E. Schilling, Sun Oil Co., secretary; Ike H. Hartsell, Tretolite Co., treasurer and James C. Orchard of Nocor Chemical Co., trustee.

Lyle R. Sheppard addressed the meeting on pipe line corrosion. He discussed forces that cause corrosion and on the methods for controlling it. Mr. Sheppard has spent some 25 years in the corrosion development and research section of Shell Pipe Line Corp., Houston.

A. B. Campbell, executive secretary of the National Association of Corrosion Engineers was a visitor and addressed the meeting briefly.

The 37 members and guests present enjoyed a social hour and a dinner before the program.

●

CORROSION's 10-year alphabetical subject index covers the years 1945-54.

●

The 1959 15th Annual NACE Conference will be held in Chicago.

Demonstration on Metallizing Planned At Los Angeles

San Diego Section will hear a talk and see a demonstration on metallizing January 22 at Lawton's Restaurant, La Mesa, Cal.

At the November 20 dinner meeting, Ian A. McMaster, San Diego Gas and Electric Co. spoke on high temperature corrosion. He described corrosion troubles and methods used to combat them at three electric generating plants of his company.

The plants range from relatively low-temperature operation to very high-temperature steam processes. Because of the use of ocean or San Diego Bay seawater for cooling, additional problems have been encountered. Mr. McMaster said his company proposes to embark on a two-year experiment in various treatments at the low temperature generating plant.

Officers for 1958 were nominated and election ballots scheduled for mailing. The nominating committee recommended Loy L. Flor, Helix Irrigation District for chairman; Kenneth R. Christy, Federal Housing Administration for vice-chairman and Reubin C. Tullis, San Diego Water Dept. for secretary-treasurer. Don P. Armbruster, San Diego Gas and Electric Co., was chairman of the nominating committee.

Shreveport Elects Irish

Officers elected to head Shreveport Section during 1958 are L. B. Irish, Irish Engineering Service chairman; J. J. Wise, Arkansas-Louisiana Gas Co., vice-chairman; R. P. Naremore, Arkansas Fuel Oil Corp., treasurer and Grady Howell, Tube-Kote, Inc., is the secretary. E. H. Sullivan, United Gas Pipe Line Co. was elected trustee.

At the November 5 meeting, H. C. Noe, Jr., Visco Products Co., Sugar Land, Tex. spoke on "Emulsion Problems Encountered in Corrosion Treatment of Wells." There were 20 members and 10 guests present. The section meets regularly on the first Tuesday in each month at the Caddo Hotel.

L. L. WHITENECK, President

A. L. STEGNER, Treasurer

HUGH P. GODARD, Vice-President

1958-59 OFFICERS

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

New Directors Are Named for Three-Year Terms

R. McFARLAND

CHARLES G. GRIBBLE

L. C. WASSON

1957 Perminan Basin Tour Rated Best; Over 200 Participate in 3-Day Event

PERMIAN BASIN TOUR participants are seen crawling over, around and into equipment displayed during the October 23-25 Seventh Biennial Permian Basin Corrosion Tour sponsored by NACE Permian Basin Section. Besides the equipment shown here, scores of other installations were inspected and the success of corrosion mitigation efforts reviewed.

John Gannon, Texas Company, Midland, chairman of the Seventh Biennial Permian Basin Corrosion Tour said that the October 23-25 event was the best held so far. Plans are being made to hold an even larger tour in 1959.

More than 200 persons participated, visiting in chartered busses sites where oil production equipment was displayed for inspection so results of corrosion mitigation could be surveyed. Much of the equipment had been seen in earlier tour conducted by the section.

During evening sessions following inspection visits, Jack Collins, Continental Oil Co. acted as moderator for discussions concerning the equipment inspected.

New Equipment Seen

New exhibits which attracted particular attention included the experimental floating deck installation in Mitchell County. This deck in a 12 foot in diameter by 15-foot stock tank was said to be effective in reducing vapor space corrosion, minimizing tank bottom corrosion and maintaining the gravity of the oil.

Reinforced plastic pipelines were found to be in good condition. Low 250-barrel plastic storage tanks in the Howard-Glasscock Field were inspected. One had collapsed recently but other were generally in good condition except for limited seepage through the plastic. A plant doing cleaning, blasting, inspecting and coating of pipe was visited and applications of modified coal tar seen at another.

Modified Coal Tars Favored

Epoxy modified coal tar coatings generally were found to be giving good service after 21 months' exposure in the notoriously corrosive Southeast Dune Field in Crane County. A 322-barrel tank coated with this material in January, 1956 was found to be in excellent condition.

Magnolia Petroleum Company's pilot waterflood in the Pegasus Field drew close attention. Effective inhibition of well head corrosion by chemicals was exhibited as were cathodic protection of a water tank and testing equipment at a 13,000-foot injection well.

Cathodic Protection Successful

Cathodic protection was found to be generally successful in treaters, water filters and other vessels.

Some installations of plastic coated sucker rods protected by injected inhibitors also were found in good condition.

In-place cleaning and coating results in piping were found to be satisfactory in a number of exhibits inspected in the Sand Hills and Andrews areas. Cement-lined pipe and flumes in gunbarrels also were found to be giving good service.

Other equipment inspected included cast iron heating coils, a bolted steel tank with aluminum deck and nickel plated bottom, pipeline inhibitor injection systems, asbestos-cement pipe in salt water disposal systems, submerged combustion and other treatment methods in Gulf Oil Corporation's North Ward Estes waterflood, special corrosion resistant pump parts and plastic coating tubing in water injection wells.

•

NACE's Corrosion Abstract Punch Card Service issued 14,699 cards through 1957.

Campbells Are Honored by Houston Section Members

Mr. and Mrs. A. B. Campbell received an engraved silver bowl from Houston Section NACE as an expression of appreciation from the members at the December 10 dinner-dance meeting.

Following dinner a program consisting of an illustrated lecture on rockets was presented by L. F. Megow, general manager, Hahn and Clay's Steel Fabricating Division. Several members of the Rocket Research and Development Society of Houston, of which Mr. Megow is technical director, presented data on their rocket research and production. The society is composed of local high school and university students interested in rocketry.

Members were reminded of the section's Corrosion Short Course to be given at the University of Houston January 23-24.

A. W. Gilbert, supervisor of safety, Carbide and Carbon Chemicals Co., will speak on "Non-Destructive Testing" at the section's January meeting.

Southwestern Ohio Section To Discuss Cable and Pipe

New developments on underground cable and pipe protection will be the subject of a technical paper to be given at the January 28 meeting of the Southwestern Ohio Section. The Cincinnati Gas and Electric Co. will be host.

At the November dinner meeting, John A. Frank, corrosion engineer with National Distillers and Chemical Corp., gave an illustrated talk on "Corrosion Control in Distillery Operations." Corrosion problems are present in all phases of the process, he said, from handling of ground grains to bottling the finished product. He discussed methods and materials used to lessen corrosion.

The meeting attracted 19 members and 6 guests. A Christmas party scheduled for Dec. 17 was cancelled because facilities were not available.

Technical Section Format Changed in This Issue

Effective with the January, 1958 issue of CORROSION the style and size of type used to set the Technical Section has been changed. The new style uses a 3-column format, essentially the same as that used for Technical Committee Reports during 1957 and the same type as has been used to set the 3-column portions of CORROSION since January, 1949.

The change was made as an economy measure because of the increasing volume of technical material being approved for publication and to offset, in part, mounting costs of publication which have increased steadily every year since the publication was founded in 1945. The change is expected to permit increasing the volume of technical material over that published in 1957 when 852 pages appeared in the Technical Section while keeping printing costs within budget allocations.

Frink Elected to Head Southeastern Region

The following officers have been elected to serve the Southeast Region for 1958: Joseph Frink, Florida Power and Light Company, chairman; Robert D. Williams, vice-chairman; G. M. Jeffares, Plantation Pipe Line Company, secretary-treasurer; and John B. Paisley, Jr., American Tel. & Tel. Co., Ass't. secretary-treasurer. The term of John B. West, currently serving as director, does not expire until 1960.

W. A. Szymanski Is New Niagara Section Chairman

W. A. Szymanski, Hooker Electrochemical Co., Niagara Falls, N.Y., has been elected 1958 chairman of Niagara Frontier Section. Other officers named were H. A. Anderson, Carborundum Metals, Inc., Akron, N.Y. and M. Stern, Metals Research Laboratories, Union Carbide Corp., Niagara Falls, N.Y.

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*Commonly attributed to Thomas Jefferson, but never supported by document.

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SECTION CALENDAR

Jan.

7 Shreveport Section, Washington-Youree Hotel.

21 Cleveland Section. Joint meeting with The Electrochemical Society. F. L. LaQue, The International Nickel Co., Inc.

21 Chicago Section. Use of Inhibitors in the Oil and Chemical Industries.

27 New Orleans-Baton Rouge Section. Engineers Club, De Soto Hotel.

28 Southwestern Ohio Section. Underground Corrosion, Sponsored by Cincinnati Gas & Electric Co.

Feb.

4 Shreveport Section, Washington-Youree Hotel.

17 Cleveland Section. Matthew M. Braidech, National Board of Fire Underwriters.

18 Chicago Section. Properties and Uses of Epoxy Resins, H. W. Howard, Shell Chemical Corp.

24 New Orleans-Baton Rouge Section. Engineer's Club, De Soto Hotel, New Orleans.

24 Tulsa Section. Mayo Hotel, Tulsa.

25 Southwestern Ohio Section, Zionsville, Ind. Corrosion in Meters and Blowers.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1958

Mar. 17-21—NACE Annual Conference, Civic Auditorium, San Francisco, California.

Oct. 5-8—Northeast Region, Somerset Hotel, Boston, Mass.

Oct. 15-17—North Central Region. Cincinnati, Ohio.

Oct. 20-24—South Central Region. New Orleans, Roosevelt Hotel.

1959

NACE Annual Conference, Sherman Hotel, Chicago, Illinois.

Oct. 12-15—South Central Region Meeting, Denver, Col.

SHORT COURSES 1957

December 9-13—Biennial Short Course on Cathodic Protection. University of Illinois and NACE.

SHORT COURSES 1958

January 23-24—Houston Section Short Course for Pipeliners, University of Houston.

Feb. 12-14—Ninth Annual Tulsa Corrosion Short Course for Pipeliners, Mayo Hotel, Tulsa, Okla.

Feb. 13-14—Annual Short Course, Shreveport Section. Centenary College, Shreveport.

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HISTORICAL NOB HILL, San Francisco, showing the Mark Hopkins hotel (top center) and to its left the Fairmont hotel. The large building in the center is the Pacific Union Club, rebuilt after the 1906 earthquake and fire. Beside it is Huntington Park. Fronting the club and park and running between the Fairmont and Mark Hopkins hotels is California street upon which runs one of the city's cable car lines.

In the left rear of the photograph are the buildings of San Francisco's business district.

More Than 120 Authors Write Technical Papers For NACE Conference

Eighty-five papers or more by more than 120 authors are being prepared for presentation during the technical program at the NACE 14th Annual Conference. The program is divided into 16 symposia, essentially on industrial lines, four round table discussions and three educational lectures. It is the most extensive technical program arranged for any NACE meeting so far.

In addition to the presentation of technical papers more than 80 meetings of NACE technical committees will be held. These meetings usually consist of discussions, but often include presentation of technical papers contributed to the committees.

The multitude of papers necessitates holding triple simultaneous symposia Monday and Tuesday; quadruple sessions Wednesday and Thursday morning and triple sessions afternoons; and quintuple sessions Friday morning to conclude the program. The Friday morning session includes the two round table discussions on general corrosion problems and underground corrosion.

As nearly as possible under conditions prevailing, an effort has been made in scheduling the program to avoid simultaneous presentation of related technical material. This manifestly cannot be completely successful because of the numerous topics and meetings involved.

An innovation at the San Francisco meeting will be suspension of technical symposia during the afternoon of Tuesday, March 18, so everyone may visit the concurrent exhibition.

Technical symposia and committee meetings on Monday, March 17 will be held at the Fairmont Hotel. All other symposia and technical committee meetings will be at the San Francisco Civic Auditorium.

In addition to meetings of technical committees numerous other committees and subcommittees will be assembled to conduct the business of the association. They include the Inter Society Corrosion Committee, Publication Committee and its subcommittee and the several standing committees. Meetings of the board of directors will be held also.

The annual general business meeting of the association will be held beginning at 11 am, Wednesday, March 19.

Besides all of the events indicated above there will be a separate program for ladies attending the conference and an extensive social program. This latter includes the annual Fellowship Hour underwritten by exhibitors and the annual banquet at which the NACE Whitney and Speller and Young Author Awards will be presented.

Ellis

Forcht

Halbig

Troiano

Corrosion Problems of New High Performance Aircraft to Be Topic of Symposium

Airframe and skin corrosion problems will be considered during a three-paper Aircraft Industry Symposium at San Francisco. New problems created by heat from air friction on skins; new fabrication techniques involving adhesive bonded, brazed and welded assemblies and a survey of corrosion resistance of stainless steel airskins are covered.

One paper considers the hydrogen embrittlement problem associated with cadmium plating of high strength hardenable steel airframe components.

ABSTRACTS

Observations on Corrosion Resistance of High Strength Stainless Steels for Aircraft by John J. Halbig and O. B. Ellis, Armco Steel Corp., Middletown, Ohio.

Corrosion of Zirconium in Hydrochloric Acid at Atmospheric Pressure by W. E. Kuhn, Carborundum Co. Niagara Falls, N. Y.

Short term corrosion tests of zirconium subjected to different surface treatments prior to immersion in hydrochloric acid solutions suggest that film formation factors play a dominant role in its corrosion behavior. Long term tests reveal two general types of films, a monoclinic zirconium oxide and a face-centered cubic zirconium monohydride.

Oxide films are subject to failure by "breakaway" mechanism involving formation of interfacial hydride at oxide-metal interface, failure time being dependent on integrity of oxide film. Film integrity is dependent on formation history. After "breakaway" rate of loss in boiling 20 percent hydrochloric acid may range from 3 to 4.5 mils per year.

Protective hydride films are black and vary widely in degree of protection conferred. Chemically polished surfaces produce porous hydride films which offer little protection, whereas electrolytically polished specimens produce dense resistant films.

Corrosion mechanisms are described and discussed. Photomicrographs of various characteristic films are given and discussed.

Hydrogen Embrittlement of Cadmium Plated High Strength Steel by Alexander R. Troiano, Case Institute of Technology, Cleveland.

Various types of electroplated surfaces are employed on steel for protection against corrosion. With the recent widespread use of steels heat treated to very high strength levels (280,000 psi), and cadmium plated for protection, there has arisen a new problem of brittle behavior. Spontaneous brittle delayed failures, apparently associated with the hydrogen of pickling and plating, can occur at loads substantially below those that may have been sustained successfully previously and in material which shows good ductility in conventional tests. Thus a "proof stress" type of test is of little value.

An evaluation of the various mechanical tests, such as tensile, notch tensile, notch impact, sustained load, etc., are considered in the light of their ability to predict behavior. This introduces the concept of the static fatigue curve and the static endurance limit.

The action of various low temperature treatments designed to "bake out" the hydrogen will be considered in terms of mechanical data, particularly the static endurance limit.

Brief considerations of the theory of delayed failure will follow. Finally, there will be a discussion of promising experiments currently underway at the Case Institute of Technology, designed to minimize the embrittlement produced by cadmium plating.

Observations on Corrosion Resistance of High Strength Stainless Steels for Aircraft by John J. Halbig and O. B. Ellis, Armco Steel Corp., Middletown, Ohio.

Increased aircraft speeds bring increased operating temperatures due to air friction. This has brought a trend to aircraft frame components of hardenable stainless steels. There are two distinct alloy groupings of these materials: 1. Hardenable chromium grades, including alloy modifications, and 2. Precipitation hardening steels.

Compositions and properties of some of these alloys are described briefly. Corrosion performance of the widely used Armco 17-7 PH precipitation hardening alloy is compared with that of other hardenable stainless steels. Data are presented also for the new precipitation hardening stainless alloy Armco PH 15-7 Mo.

Corrosion Problems of Sandwich Constructions for Airframe Applications by B. A. Forcht, Chance Vought Aircraft, Inc., Dallas.

A resume is presented to acquaint the reader with the history of sandwich constructions in the airframe industry. Materials and processes employed for adhesive bonded, brazed and welded assemblies are explained. Problems peculiar to the airframe industry, concerned with piloted aircraft and missiles, are defined with respect to environment, logistics, and life span.

Problems confronting the airframe manufacturer with respect to the mechanism of corrosion of sandwich constructions are explained. Influence of such factors as process variables and environmental conditions are examined. Data are given to show the degree of protection that can be expected under laboratory, outdoor exposure and service conditions for various pre-bonding chemical surface treatments and externally applied organic coatings. The questions of how to detect the presence of corrosion, what degree could be tolerated and what repair procedure can be utilized are raised and some possible solutions offered.

To date, indications are that external measures such as paint films or mastic coatings provide only temporary relief. The satisfactory solution lies within the sandwich construction. To accomplish it, further investigations with respect to metal surface treatments, bonding agents, and process variables are required.

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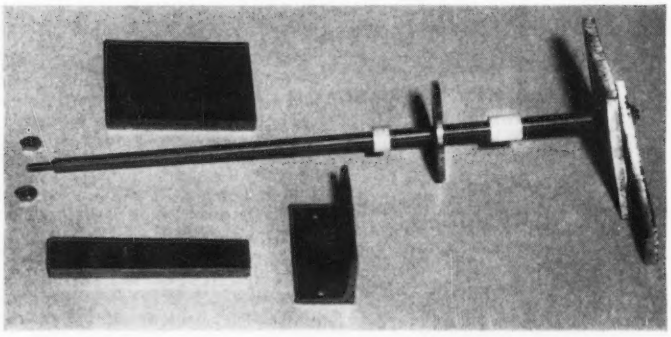

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BIOGRAPHIES

O. B. ELLIS—Senior Research Engineer, Armco Steel Corp., has a BS in chemistry, 1922 and an MS in physics, 1924 from University of Louisville, Ky. He has been in the company's research laboratories since 1932 where he supervises corrosion research.

BRENNAN A. FORCHT—Since 1951 he has been a member of the Structural Materials Dept., Chance Vought Aircraft, Inc., working on bonded structure applications. He has a MS in chemical engineering from Rensslaer Polytechnic Institute and a BS in chemical engineering from Brooklyn Polytechnic Institute. He has worked also for Socony Vacuum Oil Co. and E. I. DuPont.

JOHN HALBIG—Armco Steel Corp., Middletown, Ohio. Now senior research engineer with his company, he has been doing corrosion work since 1940, except during the war when he was with the Corps of Engineers. He presently is engaged in industrial corrosion control work, including selection of materials and maintenance procedures. His numerous activities in NACE include work on the Editorial Review Sub-Committee and numerous technical committees. He works with ASTM and AISI committees on maintenance and cleaning activities. He has a BS in chemical engineering from Johns Hopkins University, Baltimore.

ALEXANDER R. TROIANO—Head, Dept. Metallurgical Engineering, Case Institute of Technology, Cleveland. Dr. Troiano, active in research connected with phase transformations in metals, on which he has published some 50 papers, has a PhD from Harvard Graduate School of Engineering Metallurgy. He has been an instructor at Middlesex College, Mass.; at Harvard University of Notre Dame, Indiana and has held his present post at Case since 1953. His recent work has been in high strength steels and titanium alloys. He has received numerous awards and honors for his work in metallurgy, including the AIME Hunt Medal, ASM Howe medal and is active in six professional engineering organizations. He is consultant to many industrial organizations and to the U. S. armed forces.

Variety of Technical Problems Weighed In Cathodic Protection Symposium Papers

Four problems in four different cathodic protection situations will be weighed in the papers to be given during the Cathodic Protection Symposium at San Francisco. One paper tells of a successful method of installing inert anode arrays at depths up to 350 feet to protect gas pipe lines.

Another paper reviews the solution of a corrosion problem involving a pipe line paralleling a 4000-volt DC railway system with its attendant stray currents while another paper deals with protection of compressor station piping in high resistivity soil.

A fourth paper weighs the relative merits of silicon versus selenium rectifiers for cathodic protection circuits.

ABSTRACTS

Protection of Compressor Station Piping in High Resistivity Soil by Pearce R. Butterfield, Transcontinental Gas Company, Houston. Experience gained in cathodic protection of underground piping of 12 compressor stations in high resistant soils is discussed. Soil resistance from station to station varied from 25,000 ohms/cm³ to 85,000 ohms/cm³. Extensive copper grounding systems intensified the corrosion problem before application of cathodic protection and caused many spurious pipe-to-soil measurements. A comparison of the amount of current required for protection from a remote groundbed versus a distributed system within the plant is given for three stations. Geometric layout of both distributed and remote groundbeds is discussed. Construction costs of each type of groundbed are compared and the inherent operational faults explained.

Cathodic Protection of a Pipe Line Paralleling a 4000-Volt DC Railway System by L. E.

(Continued on Page 99)

Butterfield

Maddux

Tatum

Wilson

BIOGRAPHIES

PEARCE R. BUTTERFIELD—Since January 1952 he has been occupied at Transcontinental Gas Pipe Line Corp., Houston on problems associated with internal and external corrosion problems. He has a BS in civil engineering from University of Kansas and was employed by Phillips Petroleum Co. from his graduation in 1948 to June, 1950 when he started working for Trunkline on engineering in connection with construction of a gas transmission system.

L. E. MADDUX—Chief Corrosion Engineer, Continental Pipe Line Co., he joined the company at Ponca City, Okla. in 1952 after graduation from Kansas State College, Manhattan with a BS in civil engineering. He has worked as staff, design and construction engineer on several pipe line, station and terminal projects.

JOE F. TATUM—Corrosion engineer with Wilmut Gas and Oil Co., Hattiesburg, Miss. After graduation from Vanderbilt University with a BE.E., electronics major in 1945, he was employed by Westinghouse Electric Corp. in East Pittsburgh and at Sharon, Pa. as a manufacturing engineer. In 1947 he was employed by Wilmut as measurement engineer; has since done considerable work protecting bare distribution systems cathodically and holds several patents relating to pressure control, heating equipment and plugging devices. A member of a number of technical organizations, including NACE he is a registered Mississippi professional engineer and does a limited amount of consulting work.

DON P. WILSON—President of Lester Equipment Mfg. Co., Inc., he became active in the

(Continued on Page 99)

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Fink

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Chemical Industry Papers Consider Some Problems Related to Reactors, Rockets

Technical information heavily weighted on the corrosion problems associated with nuclear reactors and violently corrosive environments will be presented in a 12-paper Chemical Industry Symposium at San Francisco. Papers on the corrosion resistant properties of zirconium, titanium, columbium and molybdenum will be given along with papers dealing with such subjects as hydrogen peroxide corrosion, structural alloys in nuclear reactor cooling systems, materials for hydrochloric and nitric acid service, lead acid brick construction, inorganic cement for sulfuric acid service and others.

This is the largest chemical industry symposium to be given at a NACE national conference.

ABSTRACTS

Materials for Concentrated H_2O_2 Service by John J. Nowack, Charles W. Raleigh Food Machinery and Chemical Corp. and Ralph Bloom, Jr., Becco Chemical Division.

Reference data for use of chemical and rocket industries are presented. Fourteen years' experience in selecting, testing and evaluating metals and alloys, plastics, synthetic oils and greases and miscellaneous materials when exposed to 90 and 98 percent by weight H_2O_2 are described and discussed.

Considered are corrosion problems peculiar to materials, service classifications of materials, criteria for classifications, discussion of experience on classifications, tables and results of 66 and 30 C compatibility tests, results and problems areas that still remain. An alphabetical classification of all materials tested is included.

Corrosion Properties of Ta, Cb, Mo and W by Clifford A. Hampel, Fansteel Metallurgical Corp., North Chicago, Ill.

Corrosion resistant properties of tantalum, columbium, molybdenum and tungsten as they are affected by the nature of the corrosive medium, its concentration and temperature are presented. Tantalum is resistant to all acids except hydrofluoric and free SO_3 at normal industrial temperatures, but it is attacked by strong alkalies at room temperature. Resistance to gases depends on temperature.

Columbium generally is less resistant than tantalum to all media. Molybdenum shows good resistance to non-oxiding acids and alkaline solutions over a wide range of temperatures and concentrations and is attacked only slightly by hydrofluoric acid.

Tungsten's resistance to acids is equal to or better than that of molybdenum. All four metals are extremely resistant to liquid metals at high temperatures. Structural and fabrication limitations of each are discussed and useful applications indicated.

Corrosion Problems Associated With Uranium Refining by F. H. Meyer, F. J. Podlipec and T. R. Kato, National Lead Co. of Ohio.

This paper summarizes some of the special corrosion problems associated with the production of uranium metal from ore concentrates. After describing processing conditions and existing materials of construction, corrosion rates and effects are related to a) variable ore composition, b) alternate materials of construction, c) variable operating conditions, and d) maintenance of product purity.

Changes made in the processing system and materials of construction necessitated by the severity of corrosion are discussed.

The basic refining process is described and the performance of presently used and alternate materials of construction are compared.

Lead-Acid Brick Construction by E. J. Mullarkey, Lead Industries Association, New York.

Lead-acid brick construction is able to meet a complex variety of conditions that usually cannot be met in any other way. New test methods promote better quality and leak detection, once a serious problem, is now accomplished with the latest techniques involving isotopes and by other methods.

Two novel and informative examples of this kind of construction are given.

Conditions which lead to the selection of lead-acid brick construction are:

1. High temperatures. Lead-brick construction is used frequently where temperatures are as high as 1000 C.
2. High pressures and vacuum. Certain acid recovery units are brick and lead lined and operate under vacuums as low as 0.25-inch Hg at about 260 F.
3. Abrasion and erosion. Brickwork reduces the scouring effect of violent agitation found, for example, in sulfuric acid concentrators.
4. Thermal insulation. Essentially eliminates heat loss and excessive temperature fluctuation as often encountered under rapidly fluctuating time-temperature cycles.
5. Substantial cost savings. Corrosion resistant masonry construction can be depreciated over a long period of years, 15 years being not uncommon. Cost of new construction is considerably reduced by the scrap value of lead, and
6. Corrosion. Brick and lead perform very effectively over the range of sulfuric acid, alum and acid-alum concentrations, etc. and where various organic impurities are encountered.

A New Inorganic Cement Mortar for Sulfuric Acid Service by Robert S. Mercer, Pennsalt Chemicals Corp., Philadelphia.

Silicate cement mortars, consisting of a chemically inert solid filler containing a fluoride setting agent and a liquid sodium or potassium silicate binder have been used for many years in acid-proof construction for high temperature, strong acid service. While silicate cements have performed well under unusually rigorous conditions, they are not without their shortcomings in sulfuric acid service. Construction failures result from breakdown of the setting agent and hydrate or alum formation.

A promising new material of construction, a silica cement mortar has been developed which overcomes the shortcomings of silicate cements. The binder is a silica sol instead of a solution of a sodium or potassium silicate which is alkaline. The setting agent permits an orderly gela-

(Continued on Page 99)

Teeple

BIOGRAPHIES

W. E. KUHN—Senior Engineer directing activities of Metallurgy Section, Research and Development Div., The Carborundum Company. His activities include research and development in connection with production of zirconium and on the high intensity arc and more recently electric furnace practice. He was employed formerly by Titanium Alloy Division, National Lead Co., Niagara Falls, N. Y. At Ontario Research Foundation, Toronto, Ontario he worked on powder metallurgy techniques applicable to titanium. He received the Young Author's Prize of the Electrochemical Society in 1953 for his papers on arc melting of reactive metals. He has a BSc in metallurgical engineering from University of Toronto and is a member of numerous engineering organizations, including NACE.

W. D. MANLY—Metallurgist at Oak Ridge National Laboratories, where he does research on high temperature materials used in atomic energy work. He has a BS and MS in metallurgy from University of Notre Dame. He belongs to several engineering organizations including NACE and is a frequent contributor of technical material on his engineering specialty. He is active on NACE technical committees.

ROBERT S. MERCER—Group Leader, Technical Division Laboratory, Pennsalt Chemicals Corp., Philadelphia, he has a BS in chemical engineering, Drexel Institute. He is active in NACE as an officer of Philadelphia Section and works in technical committees A-5, 6A, 6K and 6R. He also is a member of ASTM and SPI.

FRED H. MEYER, JR.—Head of the Corrosion Section, National Lead Company of Ohio, U. S. Atomic Energy Commission Fernald Project, he has a 1949 BS in chemistry from University of Cincinnati. He started work for his present employer in 1952 and has been in charge of corrosion work since 1953. His principal activities are in connection with problems involved in refining uranium from ore concentrates and application of corrosion resistant materials of construction. He has been active in NACE technical committee work and presently is vice-chairman of T5A-5.

EDWARD J. MULLARKEY—Technical director of Lead Industries Association, he has a BS and an MS in Metallurgical engineering from New York University. He is concerned with application of lead to all phases of chemical construction and is a member of numerous engineering organizations, including NACE.

H. O. TEEPLE—General secretary of the Technical Association of the Pulp and Paper Industry Engineering Division and a member of

(Continued on Page 99)

Bigos

Munger

Palmer

Richardson

Rudolf

Whiting

Newer Plastic Systems for Severe Service Emphasized in Protective Coatings Papers

Emphasis is on the newer plastic coatings for use in severe environments during the Protective Coatings Symposium to be given at San Francisco in March. Two of the papers discuss problems of offshore drilling structures and another considers effect of anchor pattern profile on paint performance.

A survey of the use of coatings in the chemical industry and a review of wash primers and their advantages as well as a presentation of techniques for applying fluorinated plastic coatings also will be given.

ABSTRACTS

Anchor Pattern Profile and Its Effect on Paint Performance, Joseph Bigos, U. S. Steel Corp., Monroeville, Pa.

Techniques are described for measurement of the anchor pattern profile of cleaned surfaces. Effect of the cleaning techniques on surface roughness is described. Blast cleaning with high density, large particles produced excessively rough surfaces. Tests with paint were made on steel with a wide variety of anchor patterns and surprisingly, anchor pattern profile was found to have only limited effect on corrosion protection afforded by paint in atmospheric exposures.

Internal Coating of Pipe by Robert M. Johnson, Coast Paint and Lacquer Co., Houston. Data gathered in field research and in NACE committee work indicates definite trends in development of interior coatings for pipelines. No coating has been accepted as suitable for flow lines, gathering systems, salt water disposal lines and oil and gas well tubing.

Baked phenolics and epoxy-phenolics are favored, with baked phenolics having ten years of proved success and epoxy-phenolics three. Air dried coatings are being used extensively to coat lines in place. Interior lining of gas transmission pipe has increased, with mill, yard and field applied air dried coatings having up to four years of success.

Gas transmission companies use interior coatings to protect stored pipe in expectation of increased through-put, figures on which are now coming to light.

Painting for Corrosion Protection in Offshore Oil and Marine Service, Warren D. Palmer, Protective Coating Engineering and Inspection Service, Houston.

Exposures on and corrosion control on oil drilling and production equipment in the Gulf of Mexico are discussed. Importance of proper evaluation of corrosion control measures is stressed, including effectiveness of coating systems and application procedures.

Scheduling of surface preparation and coating of structures in yards and offshore are considered together with some costs. Importance of quality control, especially what is termed "industrial painting inspection" is stressed.

Though costs may seem high, presently used systems in the Gulf are having an increasingly long life, ranging from seven to as long as 20 years. It is estimated that 20-year programs cost one half or less of the original cost of the installation.

Protective Coatings for Offshore Drilling Structures by John I. Richardson and C. G. Munger, Amercoat Corp., South Gate, Cal.

Coatings used on offshore structures are considered by vehicle types, with advantages and disadvantages of each described. Coatings discussed include chlorinated rubber, vinyls, vinyl mastics, epoxies and inorganic zinc silicates. Some case histories are given, illustrated with color slides.

A Discussion of the Present Day Art of Sandblasting by H. T. Rudolf, Atlantic Coatings Co., Jacksonville, Fla.

Use and effect of air in sandblasting is detailed in terms of efficiency, velocity, temperature, pressure and field measurement. Use and differences among various types of blasting equipment is discussed with detailed reference to sandpots, hoses, nozzles, couplings and air com-

(Continued on Page 99)

BIOGRAPHIES

JOSEPH BIGOS—Since January 1, 1957 he has been with applied Research Laboratory, United States Steel Corp., Monroeville, Pa. as head of its organic coatings section. He now works on recommendations and specifications concerning maintenance painting, research on organic coatings production, on linings, containers and metallic coatings. He has a BS, MS and PhD degrees from University of Pittsburgh. With the U. S. Army 1940-46 he worked on ship repair and marine work. From 1946 through 1951 he taught chemical engineering at University of Pittsburgh, was a consulting chemical engineer 1949-50 and 1951-56 was senior fellow of Mellon Institute and director of research for Steel Structures Painting Council. Among other engineering groups he belongs to NACE.

ROBERT M. JOHNSON—Of more than 11 years spent in corrosion control work, he has been with Coast Paint & Lacquer Co., Houston more than seven years working on formulation, control and research on all kinds of coatings.

C. G. MUNGER—Vice-president in charge of manufacturing and research for Amercoat Corp., South Gate, Cal. He has been affiliated with American Pipe and Construction Company and Amercoat since 1935, starting corrosion research

(Continued on Page 99)

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Choice of Materials for High Temperature Service Covered in Diversified Papers

The extent to which the growing use of high temperature methods and processes has affected choice of construction materials is reflected in the papers to be given during the Elevated Temperature Symposium at San Francisco. Three papers deal with problems with nickel-bearing steels while a fourth discusses the effect of molten boron oxide on materials. Boron oxide's use has increased rapidly in military and industrial applications.

ABSTRACTS

Some Experiments on Internal "Green Rot" Oxidation of Nickel-Chromium Alloys by H. R. Copson and F. S. Lang, The International Nickel Co., Inc., New York.

Green rot is defined and illustrated by examples from service. This rather rare deep internal oxidation of nickel-chromium alloys sometimes is accompanied by deeper carburization or sulfidation, but in other cases these features appear to be absent.

Laboratory tests showed that when materials with less than 9 percent chromium content were exposed to air a thick external oxide was produced under which there was some internal oxidation. Increased chromium, however, reduced the attack in air to a low rate. Several unsuccessful attempts to produce internal attack in high chromium alloys are described.

It was found that exposure to carbon monoxide at 1750 F caused rapid simultaneous carburization and internal oxidation of some nickel-chromium alloys, but that addition of iron and silicon slowed the attack and addition of columbium or titanium stopped it completely. Comments are made about mechanisms, but further investigation is needed.

Corrosion Resistance of Nickel-Bearing Steels to High Temperature Lead by William Lechnick, Nuclear Dept., The Martin Co., Baltimore, Md.

Corrosion resistance of nickel-bearing steels to lead at 1400-1500 F was enhanced when nickel and zirconium were added to the lead. Materials were tested under static conditions for 340 to 5252 hours.

A description is given of test procedures and methods used to purify lead. Data on corrosion rates with and without inhibitors are presented. Included are results of microscopic examination of tested specimens.

High Temperature Oxidation of Chromium-Nickel Steels by D. Caplan and M. Cohen, National Research Council of Canada, Ottowa.

Scaling of austenitic stainless steels in air at 1600 to 2000 F has been investigated by determining weight increase with time. Curves show repeated periods of rapid oxidation. Metallo-

(Continued on Page 99)

BIOGRAPHIES

CHARLES R. ANDREWS—Projects Supervisor, Special Projects Div., University of Dayton Research Center. He has a BME, University of Dayton, 1951, and has been with the university's research center for five years. He worked for two years on development of temperature and stress measuring techniques for aircraft structures exposed to nuclear detonations and has been involved in testing special purpose materials for air weapon applications. He designed and is directing operation of the special high temperature corrosion test fixture described in his paper.

DONALD CAPLAN—In the corrosion laboratory of the National Research Council of Canada his principal interests have been corrosion of stainless steels, scaling of metals and corrosion by anti-freeze solutions. He has a Bsc and Msc from Queen's University, Kingston and a PhD in metallurgy, Rensselaer Polytechnic Institute, 1956.

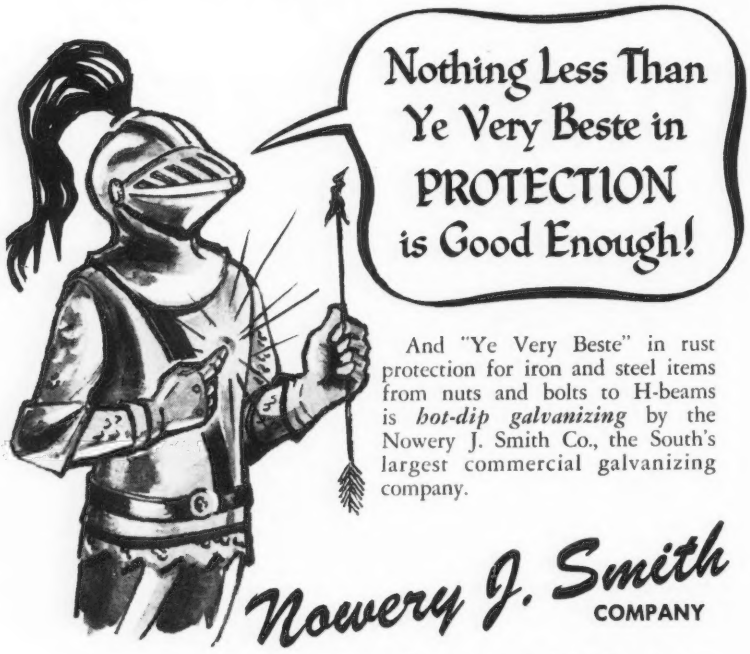
MORRIS COHEN—After experience as a chemist at a battery manufacturing plant, he joined National Research Council of Canada, Ottawa. He has been head of the Corrosion Section, Division of Applied Chemistry since 1946. He has a PhD from University of Toronto, 1939 and has authored numerous articles on corrosion in aqueous solutions and on high temperature oxidation.

FRANCES S. LANG—With International Nickel Co., Inc., since 1951, she has a PhD in physical chemistry, New York University, 1952. She is research chemist, Corrosion Section, Research Laboratory, Bayonne, N. J.

DAVID ROLLER—Project Engineer, Wright Air Development Center, Materials Laboratory, Wright-Patterson AFB, Ohio. He has a BS, chemistry from Massachusetts Institute of Technology, 1950 and has worked for Freeport Sulphur Company on problems associated with nickel-cobalt ores. His work with the air force has been principally in the corrosion field. Among other engineering organizations, he belongs to NACE.

WILLIAM LECHNICK—A corrosion engineer in the Corrosion Group, Nuclear Division, The Martin Company since January, 1957, Mr. Lechnik formerly worked in the chemical engineering division, Argonne National Laboratory where he became familiar with corrosion problems encountered in working with fluorine compounds. He has a MS in chemistry, 1952, Aurora College.

Quoth Sir Galva-Knight:



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Educational Lectures Cover Design and Corrosion Reactions

Mechanisms of corrosion of ferrous and non-ferrous metals, always subjects of primary interest, will be covered during one of the three educational lectures to be presented during the 14th Annual NACE Conference at San Francisco. Also scheduled are lectures on design and interpretation of atmospheric corrosion experiments and a review of the problems involved in deterioration of materials in polluted atmospheres.

Educational lectures have been among the best attended sessions during NACE technical programs. All speakers have had long experience in their subjects.

ABSTRACTS

Deterioration of Materials in Polluted Atmospheres by John E. Yocum, Bay Area Pollution Control District.
A brief summary of the chemical and physical aspects of polluted atmospheres. More specifically data on the detrimental effects of air pollutants on materials other than living tissue. In addition to corrosion of metallic surfaces, physical and chemical principles involved in damage to such materials as paint, glass, stone, rubber and fibers by man-made air contaminants are discussed.

Mechanisms by Which Ferrous Metals Corrode in the Atmosphere by C. P. Larrabee, Applied Research Laboratory, United States Steel Corp., Monroeville, Pa.
The electrochemical theory of corrosion, by which the corrosion of steels in solutions is readily explained, also can be used to explain the start of atmospheric corrosion on ferrous surfaces. The effect of contaminants both in the atmosphere and on the steel surface is described. Experiments leading to this conclusion are illustrated.

The part played by various alloying elements in retarding atmospheric corrosion, once a rust film is formed, will be discussed. The reasons that certain elements do not permit the rust films to become protective also will be discussed.

Mechanisms by Which Non-Ferreus Metals Corrode in the Atmosphere by P. M. Aziz and Hugh P. Godard, Aluminium Laboratories, Lt. Kingston, Ont.
Literature dealing with the mechanism by which non-ferrous metals corrode in the atmosphere is reviewed, particular emphasis being given to the important series of papers published by W. H. J. Vernon and his co-workers from 1923 to the present. Influence of common atmospheric constituents is discussed and related to the chemical composition and properties of the corrosion produced film formed. Influence of these on the atmospheric corrosion rate is discussed also in relation to the nature of the corrosive atmosphere. Atmospheric corrosion of aluminum, brass, copper, lead and zinc is discussed.

Principles and Procedures Employed in Design and Interpretation of Atmospheric Corrosion Experiments by H. R. Copson, International Nickel Co., Inc., Bayonne, N. J.

Yocum

BIOGRAPHIES

P. M. AZIZ—He is concerned principally with basic research on the corrosion mechanisms of aluminum on which he has published six papers. He holds a BASc in chemical engineering and MA and PhD degrees in physical chemistry from the University of Toronto where he studied surface tension in aqueous sulfuric acid solutions and transference phenomena in molten salts. He has been on the staff of Aluminium Laboratories, Ltd., Kingston, Ont. since 1949. He spent a year at University of Chicago developing a radiotracer technique for identifying cathodic sites on aluminum surfaces. He was the recipient of the 1945 NACE Junior Award for the best corrosion paper published in CORROSION by an author under 35 years of age.

HARRY R. COPSON—Head of the Corrosion Section, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J. he has been active in engineering associations for many years in organizing and managing corrosion control work. He is a member of many technical committees, has authored numerous papers and has a PhD from Yale University, 1932.

HUGH P. GODARD—Recently elected vice-president of the National Association of Corrosion Engineers, he has been active in NACE for several years as organizer of Canadian region and a member of the board of directors while doing research principally on the corrosion reactions of aluminum. He is a member of several Canadian technical organizations. He has a PhD from McGill University and is author of several papers, principally on fundamental corrosion reactions. He has been with Aluminium Laboratories, Ltd., Kingston, Ont. since 1945.

C. P. LARRABEE—Formerly chairman of the Technical Practices Committee of the National Association of Corrosion Engineers, a member of the board of directors and active in the association in numerous other ways, Mr. Larrabee has spent mre than 20 years in charge of corrosion work in the Research and Development Laboratory of The United States Steel Corp., Monroeville, Pa. A graduate of the University of Maine, he spent 10 years with the National Bureau of Standards before joining United States Steel. He is a frequent contributor of technical material on corrosion reactions.

JOHN E. YOCUM—Director of Technical Services, Bay Area Air Pollution Control District. He has a BSc, Massachusetts Institute of Technology, 1947, and for nearly 10 years worked at Battelle Institute as a project leader in charge of air pollution field surveys, designing air pollution abatement equipment and developing instruments to measure pollution. He presently is setting up a laboratory for the district.

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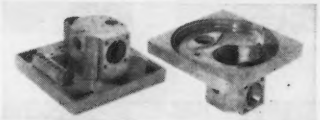

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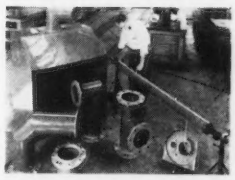

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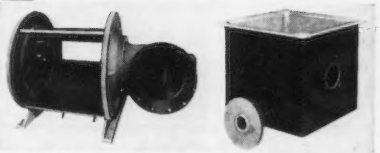

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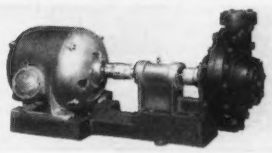

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General Symposium Topics Include Steel For Marine Service, Tank Trucks, Stress

Diversity of subject matter characterizes the 10 papers to be given during the General Corrosion Symposium at San Francisco. Subjects include steel for marine service, sigma phase effects in stainless steels, titanium, stress corrosion in aluminum bronze, metals in tropical environments, influence of temperature on aluminum, corrosion problems associated with tank trucks, ductile iron and tin plated steel pickling tests.

The stature of the authors of these papers, as well as their subject matter is sure to attract heavy attendance. This symposium in the NACE Conference technical program has been popular every year.

ABSTRACTS

Corrosion Problems of Tank Truck Transportation by Louis Reznek, National Tank Truck Carriers, Inc., Washington, D. C.

Corrosion problems of the for-hire tank truck industry in its operation of about 26,500 tank vehicles is covered. General types of vehicles are described and shown in illustrations including tanks for transporting flammable and corrosive liquids and compressed gases. Materials of construction discussed include various types of steel, aluminum. Coatings on these materials are considered.

Tanks are being used now to transport over 500 commodities at wide ranges of temperature, viscosity and corrosiveness. A brief outline of these commodities is given.

Discussed also are conditions of tank truck operation which affect corrosion rates, especially those that differ from those of fixed plant operations." Included are: Large percentage of empty "time," wide variety of products transported in one tank, effect of prior products in combination with present product and methods of cleaning on corrosion, inability to clean at time of unloadi and problems of contamination of product .y slight degrees of corrosion.

Also discussed are eight recent corrosion problems ranging from the transportation of wine and hydrofluosilicic acid to the effect of brine solutions on city streets on aluminum tanks with steel under-carriages.

The industry's methods of attacking its corrosion problems by selecting materials, linings, using sprays, cleaning methods and correct design are also discussed.

Corrosion Resistance Characteristics of Ductile Iron by F. L. LaQue, The International Nickel Co., Inc., New York, N. Y.

The internal structure of ordinary grey iron, pearlitic ductile iron, ferritic ductile iron, malleable iron and mild steel are compared. Differences in the amount of carbon present and in the form in which it occurs are responsible for some differences in the corrosion behavior of these materials. This is explained on the basis of the electrochemical reactions which occur during corrosion. As corrosion proceeds, the graphite which is present in the cast materials may accumulate as a graphitic layer on the surface. The adherence and permeability of such graphitic layers and their influence on subsequent corrosion and on galvanic behavior are discussed. Corrosion data in acids, in neutral and alkaline solutions, in sea water, in the atmosphere and underground, in miscellaneous environments and under conditions involving erosion are presented and interpreted in the light of the principles outlined above. By and large, it is concluded that ductile iron has satisfactory corrosion resisting properties which permit its use where grey iron, malleable iron or steel are regularly employed.

Factors Influencing the Rate-of-Pickling Test on Tin-Plate Steel by R. M. Hudson and G. L. Stragand, U. S. Steel Corp., Applied Research Laboratory, Monroeville, Pa.

"Lag time" is a measure of the time of pickling necessary to produce a constant rate-of-weight loss from steel immersed in acid. This measurement has been used as a guide for improving the corrosion resistance of commercial electrolytic tin plate. It is determined by measuring either the rate of change of weight loss, hydrogen evolution, or corroding potential of a specimen in 6 N hydrochloric acid at 90 C (194 F.)

Lag time depends on surface effects inasmuch as removing the surface layers of steel by abrasion or by pickling destroys the lag. Influence of box-annealing atmospheres, cleanliness of steel and time-temperature cycles on lag time has been investigated and the complexity of these effects has been demonstrated. Explanations in terms of oxidation or decarburization of the steel surface during annealing are not

(Continued on Page 97)

BIOGRAPHIES

ALLEN L. ALEXANDER—Acting Associate Superintendent, Chemistry Div., Naval Research Laboratory and head of the laboratory's protective coating branch, he holds BS, MS and PhD degrees from University of North Carolina. He is active in several engineering organizations, has been chairman of Baltimore Section NACE and has participated in NACE technical programs. He also is a member of the CORROSION Editorial Review subcommittee.

H. B. BOMBERGER—Senior research metallurgist for Rem-Cru Titanium, Inc., Midland, Pa. He has been engaged in solving corrosion problems for more than five years. He has a BS, 1942, Pennsylvania State University, was employed at Wright Aeronautical Corp. and served in the army before receiving MS and PhD degrees from Ohio State University, 1950-52.

E. H. DIX, JR.—Assistant Director of Research, Alcoa Research Laboratories, Aluminum Company of America, New Kensington, Pa. His principal technical interest is in aluminum and magnesium alloys, with special attention to their resistance to corrosion and stress. He is a member of numerous engineering organizations, including NACE, from whom he has received many honors including the 1954 NACE Speller Award for achievements in corrosion engineering and the Saveur Achievement award from ASM for his work in aluminum alloy research.

B. W. FORGESON—Since 1953 has been in charge of tropical exposure and laboratory facilities in the Panama Canal Zone for Naval Research Laboratory. He has studied corrosion, protective coatings and biological deterioration of materials. In 1940-52 he worked for Special Engineering Division of the Panama Canal as a materials engineer on the plans for construction of a third set of locks and a sea level canal. He attended Pennsylvania State University and is a NACE member.

R. M. HUDSON—Section Head, Physical Chemistry Section, Chemical-Metallurgy Division Applied Research Laboratory, United States Steel Corp., Monroeville, Pa. He has a BS in chemistry, 1947, and a PhD in chemistry from Yale in 1950. His technical experience includes work in electrochemistry, gas-metal reactions during heat treatment of steel, corrosion studies on tin plate and hydrogen behavior in steel.

JOHN F. KLEMENT—Technical director, Ampco Metal, Inc., since 1951, he formerly was chief metallurgist and wrought products superintendent. He has a BSME, Marquette University, 1940.

F. L. LaQUE—Vice-president of The International Nickel Co., Inc., New York and man-

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High Purity Water Engineering Problems In Reactors and Boilers Are Covered

Growing interest in water cooled nuclear reactors and the ever increasing temperatures and pressures of steam power plants makes the subject matter of the High Purity Water Symposium of pressing interest. In this series of papers, NACE continues its contributions to the store of knowledge concerning reactions of materials to high purity water.

Behavior of zirconium-uranium alloys is weighed in one presentation, while another discusses the engineering problems solved in the first Army Package Power Reactor.

Berry

Williams

ABSTRACTS

Corrosion and Water Purity Control for the Army Package Power Reactor by Richard J. Clark and A. Louis Medin, Alco Products, Inc., Schenectady, N. Y.

The Army Package Power Reactor, a 10 MW heat rated pressurized water system designed to produce 2000 KW, has been in operation since April, 1957. Design problems associated with corrosion and water purification are presented. Primary purification system is described and performance data given on individual components. The 2-cubic-foot demineralizer has performed satisfactorily, consistently maintaining coolant resistivity above 600,000 ohm/cm. Primary coolant pH has varied between 6.4 and 9.0, depending on primary system in-leakage. Purification system has required minimal attention and has self-regulating hydrogen control. Experimental in-plant equipment for determining crude levels, corrosion rates and other related parameters is described. Preliminary studies indicate corrosion rate of system components has been lower than the originally estimated 0.05 mg/cm²/mo.

Corrosion Behavior of Zirconium-Uranium Alloys in High Temperature Water by Warren E. Berry and Robert S. Peoples, Battelle Memorial Institute, Columbus, Ohio.

The effect on corrosion behavior of water temperature, uranium content, and oxygen content has been determined for zirconium 7 through 70 w/o uranium alloys. Corrosion rates increased as water temperature increased from 500 to 680 F, as uranium alloy content increased and as oxygen content in the alloys increased. The amount of distribution of epsilon phase present in alloys containing up through 55 w/o uranium determined their corrosion behavior. Increase in corrosion rate with increased oxygen content correlated well with the effect of oxygen in forming structures having alpha zirconium and alpha uranium in the epsilon.

Removal of Corrosion Products from High Temperature, High Purity Water Systems with an Axial Bed Filter, by S. L. Williams and R. E. Larson, Knolls Atomic Laboratory, Schenectady, N. Y.

Concentration of corrosion products and other impurities which produce undesirable radionuclides must be maintained at extremely low values in the primary coolant systems of nuclear power plants to keep the resulting does rate below maximum values. This has been accomplished previously by low temperature ion exchange of the pressurized water coolant.

Development of an axial bed filter which can remove radioactive corrosion products from high purity pressurized water coolant at operating temperatures is described. It can be used also to maintain coolant pH above 8.5 at operating temperatures up to 450 F.

Some Relations Between Deposition and Corrosion Contamination in Low Makeup Evaporator Systems for Steam Power Cycle, Henry Kehmna and Elmer Johnson.

ABSTRACTS

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feasible for the development of lag time under all the experimental conditions studied.

Preliminary data demonstrating the high concentration of certain elements on the steel surface before annealing and the enrichment of the surface layer by some of these elements during annealing, are suggested as particularly promising areas for future work, so that lag-time phenomena in tin-plate steels may be better understood.

Some Observations on Stress Corrosion Crack Paths in Alpha Aluminum Bronze in Ammonia and Steam Atmospheres by J. E. Klement, R. E. Maersch and P. A. Tully, Ampco Metal Inc., Milwaukee.

The investigation presented illustrates the importance of analyzing stress corrosion crack paths and the interpretation of the crack paths with respect to alloys and corrosive media. Testing involved alpha aluminum bronze alloys of from 5% Al to 8% Al content exposed under stresses of about 80 percent of the yield strength to ammonia and steam atmospheres. Various crack paths obtained are illustrated in micrographs and the reasons for the different types of crack paths associated with factors such as equilibrium grain boundary segregation and the chemical attack of corrosive agents on segregated solute elements. Results of the investigation are discussed in terms of analyzing causes for field failures. Examples of processing considerations to minimize failures are cited.

Corrosion of Metals in Tropical Environments. Part 2—Corrosion Characteristics of Structural Steels During Atmospheric Exposure to Tropical Environments by C. R. Southwell, B. W. Forgeson and A. L. Alexander, U. S. Naval Research Laboratory, Washington, D. C.

As a continuing part of an extensive investigation of the corrosion characteristics of a wide variety of metals in the topics, the present report presents data on the corrosion rates of ten structural steels exposed to seashore and inland environments in the Panama Canal Zone. This, the second report in this series, presents eight-year exposure data on the ten steels, which permits the establishment of significant time-corrosion curves for these metals. Inasmuch as similar data exist for several of these steels during exposure to atmospheric environments in temperate zone, a basis is presented for comparing rates in the two latitudes.

Titanium, the Chemical Industry's New Material of Construction by A. G. Caterson, H. B. Bomberger, Jr., and J. D. McDonough, Rem-Cru Titanium, Inc., Midland, Pa.

Development of titanium prototypes for corrosion resistant applications is discussed to illustrate the fact that titanium is rapidly becoming a standard material of construction in the chemical industry. Attention is focused on comparative costs with special emphasis on titanium's longer service life; the reduction of down-time and maintenance costs and the steady decline in the initial cost of titanium.

A number of case histories are reviewed which demonstrate the superior service life and corrosion resistance of titanium when subjected to attack by sodium hypochlorite, nitric acid, calcium hypochlorite, ferric chloride, sulfuric acid and chlorine dioxide.

Effect of Sigma Phase vs Chromium Carbides on the Intergranular Corrosion of Types 316 and 316L Stainless Steel by Donald Warren, E. I. duPont de Nemours & Co., Inc., Wilmington, Del.

A study was made of the intergranular corrosion behavior of Types 316 and 316L stainless steel in twelve different acid environments. Included in these environments were nitric, nitric-hydrofluoric, acetic, citric, sulfuric, lactic, oxalic and phosphoric acids. Results of this study showed that sensitized and welded Type 316L stainless steel had excellent resistance to intergranular attack in all of the acid media except nitric. This was true despite the presence of a continuous network of sigma phase in the grain boundaries. In contrast, Type 316 stainless steel containing a continuous grain-boundary network of chromium carbides underwent severe intergranular corrosion in ten of the twelve environments studied.

Influence of Service Temperature on the Corrosion Resistance of Wrought Aluminum-Magnesium Alloys by E. H. Dix, Jr., Aluminum Company of America, New Kensington, Pa.

BIOGRAPHIES

WARREN E. BERRY—Project leader, Corrosion Research Division, Battelle Memorial Institute, Columbus, Ohio. He is a graduate of Ohio University, 1947 and has taken postgraduate work at Ohio State University. His work includes high temperature and high pressure corrosion reactions, fuel-element corrosion and materials for reactor construction. He has been working in the Battelle AEC reactor program since 1949.

R. J. CLARK—Senior Chemical Engineer, Alco Products Atomic Energy Department for the past two years, he has been working on corrosion and water treatment aspects of nuclear power plant design. He was employed for more than four years at the duPont Savannah River Plant, where his experience included heavy water technology, reactor operation, fuel reprocessing, fabrication and health physics. He is a graduate of Rensselaer Polytechnic Institute.

R. E. LARSON—Research Associate, Knolls Atomic Power Laboratory, General Electric Company for the past nine years, he has been concerned with coolant corrosion problems in sodium and water cooled propulsion and production type reactors. His experience is extensive on design, chemical development, radioactive maintenance and pilot plant and test facility operation. He formerly worked on Manhattan Project at Oak Ridge and University of Chicago. He has a BChE, University of Minnesota and MS, Lafayette College. He belongs to ACS and AIChE.

A. LOUIS MEDIN—Chief of the Chemical Technology Division, Alco Products' Atomic Energy Dept. for the past three years, his primary responsibility has been for all chemical and engineering aspects of nuclear power plant design and operation. He directs research and development programs in corrosion, water treatment, fuel reprocessing, waste disposal and radiochemistry. He worked formerly for Ford Motor Co. and USAEC. He has a BS, Johns Hopkins' and PhD, Ohio State University.

SHERMAN L. WILLIAMS—Concerned with process design and coolant problems associated with nuclear power plant development at Knolls Atomic Power Laboratory. He has attended Cambridge, has a BS in chemical engineering, Iowa State College and MS, Rutgers, 1951.

Crevice Corrosion in Titanium by D. R. Zaremski, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

Corrosion-Resistant Experimental Steels for Marine Applications by C. P. Larrabee, United States Steel Corp., Monroeville, Pa.

TURN TO PAGE 75 for Technical Committee Meeting Schedule

Cartledge

Henry

Hurd

Piehl

Schwoegler

Snavely

Stedman

Thompson

Wankat

Use and Effects of Inhibitors Under Varied Conditions Covered in Symposium

Use and effects of metallic and organic inhibitors under a wide variety of conditions are considered in the Inhibitor Symposium to be given at San Francisco during the 14th Annual NACE Conference. Such subjects as the influence of ionic state on certain types of inhibitors, special ways to evaluate inhibitors used in petroleum refineries and use of organic and oil soluble inhibitors will be considered.

ABSTRACTS

Inhibition of Corrosion of Mild Steel in NH_4NO_3-NH_3-H_2O Solutions by Norman Hackerman, Ray M. Hurd and Earl S. Snavely, Jr., University of Texas, Austin.

Corrosion rates of mild steel in mixtures of NH_4NO_3-NH_3-H_2O were measured at temperatures of 30, 45 and 60 C. Effects of various sulfur and arsenic-containing inhibitors were studied, with particular emphasis on NH_4CNS. The corrosion reaction was found to be markedly affected by stresses in the metal to the extent that completely stress-relieved coupons would not corrode at all. The rate of the uninhibited reaction was much lower at 45 and 60 C than at 30 C, indicating a change in the mechanism of the corrosion reaction.

The uninhibited corrosion rate of 3700 mdd at 30 C was decreased to approximately 25 mdd with NH_4CNS concentrations greater than 0.1 percent. At 45 C, the most effective inhibitor was found to be 0.05 percent 2-mercaptoethanol + 0.05 percent sodium arsenite, followed closely by 0.1 percent NH_4CNS + 0.05 percent sodium arsenite. Thiocyanate and thiourea alone at concentrations of 0.1 percent were somewhat less effective.

Simple potential measurements using a platinum reference electrode showed the corroding coupons to be about 0.9 volt more negative than the stress-relieved coupons.

Action of the XO_4^{n-} Inhibitors by G. H. Cartledge, Oak Ridge National Laboratory, Oak Ridge, Tenn.

Experimental studies on the effect of ions and molecules of the type XO_4^{n-} upon the corrosion of iron and steel will be summarized. Measurements of electrode potentials in inhibited solutions and the effect of foreign electrolytes upon these potentials have shown the competitive and reversible nature of the surface state associated with inhibition. Studies with the pertechnetate ion as inhibitor have demonstrated the absence of detectable continuing chemical action in the systems over a period of almost five years. It was shown that the passivation by OsO_4 is so complete that an iron electrode indicates the thermodynamic potential of the Os^{IV}-Os^{VIII} couple. It was also discovered that the Flade potential may be observed on iron electrodes in aerated, inhibited solutions, without use of such vigorous passivating agents as HNO_3 or anodic polarization. Results are discussed by considering the ways in which the presence of an adsorbed inhibitor may affect the kinetics of electrode processes at a metal-solution interface. It is shown that specific differences among the XO_4^{n-} particles with respect to inhibition may arise from the nature of the electrostatic field exerted by such particles over short distances.

Evaluation of Some Organic Corrosion Inhibitors for Special Applications in Petroleum Refining by R. B. Thompson, R. F. Stedman, Charles Wankat and R. C. Henry, Universal Oil Products Co., Des Plaines, Ill.

Evaluation of corrosion inhibitors used in various phases of refining is difficult in the laboratory. Some inhibitors with an extensive record of successful operation in various places in the refinery—in crude distillation columns for example—are ineffectve when tested by the ASTM Turbine Oil Corrosion Test or by the static water drop test. So, an effort was made to develop a test method to evaluate corrosion inhibitors for use in such places as crude distillation columns.

Apparatus has been devised for laboratory use to evaluate oil and water soluble corrosion inhibitors for steel at an acid pH and Admiralty metal corrosion inhibitors at an alkaline pH. Essentially, this apparatus consists of a condensing system which allows a mixture of water and hydrocarbon to flow over the strip in the presence of corrosive agents such as are normally found in a particular environment, and, essentially in the absence of air.

Reasonably reproducible results have been obtained with water soluble inhibitors on steel at mildly acid pH. Reproducibility is poorer in the case of corrosion of Admiralty metal at a slightly alkaline pH. However, it has shown that a number of proprietary corrosion inhibi-

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BIOGRAPHIES

MAURICE R. BARUSCH—Senior Research Chemist, California Research Corp., Richmond, he has AB and PhD degrees in chemistry from Stanford University. He is author and co-author of papers on subjects related to hydrocarbon combustion and has many patents on engine fuel additives.

G. H. CARTLEDGE—Since 1951 in the Chemistry Division, Oak Ridge National Laboratory. He has had many years of academic work including professorships at Johns Hopkins and University of Buffalo, has served as head of the materials department, Curtiss Wright Research Laboratory, Buffalo and is author of several textbooks on inorganic and physical chemistry as well as many research papers. He was educated at Davidson College and University of Chicago.

L. G. HASKELL—Chief engineer, Salt Lake Pipe Line Co. With a BA in civil engineering, Stanford University, 1921, his entire professional experience has been with Standard Oil Company of California and its subsidiaries. His work includes design, construction and operation of oil pipe line systems. He is vice-chairman of Salt Lake Section NACE.

R. C. HENRY—Presently engaged in corrosion and stability testing and analytical research for Universal Oil Products Company, which he joined in 1953, he previously had four years of experience as chemical officer with the 12th and 14th Army Air Force and did organic functional group analytical research for Shell Development Company. He has a BSc, in chemistry, University of Maryland, 1942; MS in chemistry, University of Michigan, 1951. In 1952 he helped set up the process control laboratories at the Paducah Gaseous Diffusion Plant of the AEC.

RAY M. HURD—Supervisor of Physical Chemistry Section, Defense Research Laboratory, University of Texas, Austin. He formerly was research chemist, Atlantic Refining Co., Dallas. He has a BA, 1943, University of Oklahoma; BS, 1949, in chemical engineering and a PhD in chemistry, University of Texas, 1954.

ROBERT L. PIEHL—Materials Engineer, Standard Oil Co. of Cal., at Richmond He has a BS in chemical engineering, 1953, University of Wisconsin and since has been concerned with corrosion in petrochemical and petroleum industries. He is chairman of the program coordination committee for the 1958 NACE 14th Annual Conference.

EDWARD J. SCHWOEGLER—Senior Scientist, Armour Research Foundation, he has BS and PhD degrees in organic chemistry from University of Wisconsin. His interest and experience includes corrosion inhibitors, hydrogenation, catalysis, fungicides, pharmaceuticals and petrochemicals. His past affiliations include University of Wisconsin, Pennsylvania Salt Co., Wyandotte Chemicals, Sinclair Refining Co. and the Daubert Chemical Company. He has ten patents and numerous publications to his credit.

EARL S. SNAVELY, JR.—Research Chemist Defense Research Laboratory, University of Texas, Austin. He formerly did chemical research for Texas A and M Research Foundation and Columbia-Southern Chemical Corp. He has a BS, 1947 in chemistry, Texas A & M College and MA, chemistry, University of Texas, 1950.

RUSSELL F. STEDMAN—A member of the Products Sales Dept., Universal Oil Products Co., Des Plaines, Ill. He has a BSc in chemistry from University of Illinois, 1935. He formerly was chief research chemist with Cardox Corp., Chicago and with Leonard Refineries, Inc. He is a member of NACE.

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ABSTRACTS

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Maddux, Continental Pipe Line Co., Ponca City, Okla.

Technique used to eliminate a severe case of external pipe line corrosion being caused by stray current is described. The products pipe line in question closely parallels a 4000-volt DC electrified track system for approximately 32 miles, wherein the two structures cross six times.

Fixtures were required at certain points to supplement and function in conjunction with conventional type cathodic protection rectifiers in order to effect 100 percent cathodic protection on the pipe at all locations throughout the stray current area. The fixtures performed efficiently as electric "check valves" for all conditions of track loading when connected between rail and pipe.

Efficacy of the technique employed is illustrated by 24-hour recorded charts of performance, showing the unprotected pipe was reversed in polarity and that the extremely severe corrosion was entirely eliminated.

Impressed Current Anodes Installed and Backfilled at Depth of 350 Feet by Joe F. Tatum, Willmut Gas & Oil Co., Hattiesburg, Miss.

Where power costs are high and low resistance soil is hard to find, a solution is to install high silica cast iron ground anodes and backfill them with coke breeze or graphite at depths of 350 feet. Method is described in detail. Type of wire used, header wire sizes, method of connection and insulation protection are described. Resistances of 1 ohm to ground are obtainable readily by this method with soil resistances on the order of 2000 ohms per centimeter. Gas blocking has not occurred with a 10 to 20 ampere drain. Installations are a year old. Accrued benefits, such as improved current distribution, low interference effect and installation savings are discussed. Protection is extended to approximately 40,000 square feet of poorly coated pipe in a half-mile radius of the groundbed.

Field Service Results of Silicon vs Selenium in Cathodic Protection Rectifiers by Don P. Wilson, Lester Equipment Mfg. Co., Inc., Los Angeles, Cal.

The high conversion efficiency of silicon rectifiers for DC output ratings above 30 volts has resulted in substantial savings in power costs. However, silicon failures in high lightning incidence areas and on power supply lines with poor control of switching transients have been unacceptably high. Peak inverse voltage failures of silicon can be caused by lightning induced transients in either the supply lines or in the CP loop without a direct strike. Silicon and selenium failure rates by areas are compared with a national Bureau of Standards lightning incidence rate map. Data covering the variation of CP loop resistance from dry anode bed to moist soil condition confirms the wisdom of secondary over-current protection. A magamp circuit which controls DC current within plus or minus 2 percent with loop resistance varying from 0.6 to 2.5 ohms is discussed. Present and future developments to protect silicon against PIV failures are discussed.

BIOGRAPHIES

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cathodic protection field shortly after joining Lester in 1955. He was among the first to employ silicon rectifiers and was identified with their application to well casings in the Shell-Ventura field. A graduate in aeronautical Engineering at Boeing School of Aeronautics, he has been employed in United Airlines, Fairchild Aviation Corp., Federal Telephone and Radio and while on active duty in the navy did work connected with airline operations, navigation and pilot and aviation radio equipment. He set up the West Coast Division selenium manufacturing operations for Federal Telephone and Radio.

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tion of silica resulting in a strong mortar containing 98% SiO_2. Physical strength and chemical resistance data are given, and its superior nature is explained theoretically.

Resume of Corrosion Characteristics of Structural Alloys in Various Coolants by W. D. Manly, Oak Ridge National Laboratory, Union Carbide Nuclear Co., Oak Ridge, Tenn.

Engineering Use of Sodium as a Heat Transfer Agent and Its Corrosive Effect by W. D. Manly, Oak Ridge National Laboratory.

Control of Corrosion in Nitric Acid Solutions by C. L. Patterson and F. W. Fink, Battelle Memorial Institute, Columbus, Ohio.

Materials of Construction for Hydrofluoric Acid Service by F. W. Fink, Battelle Memorial Institute, Columbus, Ohio.

Corrosion in the Production and Handling of Fertilizer Chemicals by H. O. Teeple, The International Nickel Co., Inc., New York.

Corrosion of Zirconium in Hydrochloric Acid at Atmospheric Pressure by W. E. Kuhn.

Titanium Equipment and Corrosion Economics by G. T. Bedford.

Stress Corrosion of a Stainless Steel in Dowtherm by D. K. Priest, Pfaudler Co., Rochester, N. Y.

BIOGRAPHIES

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the Tappi Corrosion Committee. He joined International Nickel Co., Inc., in 1944 as a member of the corrosion engineering section after working on materials specifications in plant corrosion problems at Westvaco Chemical Division, Food Machinery and Chemical Corp., South Charleston, W. Va. He has a BS ChE, 1937, University of Michigan. Among the fields in which he has specialized are the pulp and paper industry. He is a member of Canadian Pulp and Paper Association, The Electrochemical Society and American Chemical Society, among other organizations.

ABSTRACTS

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pressors, together with economics involved. Quality of available U. S. sand is considered and advantages and disadvantages of various sand sizes discussed. Practical techniques in blasting to get best efficiency at least cost are covered in detail. Specifications relating to surface cleanliness are generally discussed and some recommendations made for improvements in specifications writing.

Chemical Process Industry's Use of Plastics for Corrosion Protection, prepared under direction of W. J. Sauber, The Dow Chemical Co.

In-plant experience is an essential suppliment to engineering data designed to make successful the use of plastics in preventing corrosion. Case histories are presented describing application of epoxies, Saran, polystyrene and polyethylene in brine and acid handling systems, cathodic insulation and packed columns. Analysis of experience under a wide range of environments and conditions provides improved basis for successful use of plastic materials.

Wash Primers, by L. R. Whiting, Bakelite Company, Division Union Carbide Corp., New York, N. Y.

Development, characteristics and properties of wash primers are explained. Data are given on wash primers principally because of their efficiency and wide use. Reactive and non-reactive types resulted from efforts to understand better the reactions involved and as a result of efforts to produce a one-package system.

Reacted wash primers are made by reacting phosphoric acid and chromium oxide in a solution of buytral resin at high temperature, producing a clear green lacquer. Non-reactive primers are prepared by dispersing chromates of lead, strontium and barium in a solution of butyral resin and then adding phosphoric acid. A stable wash primer is prepared by dispersing chromic phosphate in a solution of PVB.

Kel-F Coatings by Fred Troester, Minnesota Mining & Mfg. Corp., Los Angeles, Cal.

BIOGRAPHIES

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investigating deterioration of concrete from sewage. For the past 20 years he has occupied himself with application of various plastics as coating and lining materials, working with the armed forces and the Atomic Energy Commission in solution of corrosion problems. Long active in NACE in local, national and tecnhical committee affairs, he is a frequent speaker at NACE-sponsored technical sessions. He presently is a director representing corporate members.

WARREN D. PALMER—Consultant and head of Protective Coatings Engineering and Inspection Service, Houston, he specializes in application of protective coatings in controlling industrial and marine exposures. After studying mechanical engineering with Kansas State College, Manhattan he was employed at Monsanto Chemical Company's Texas City plant from 1942 to March 1954. At Monsanto, beginning in 1948 he worked with the corrosion engineering section as inspector and protective coating engineer. He also has been employed as a field engineer for The Austin Company and for Ford, Bacon and Davis.

JOHN I. RICHARDSON—For more than 16 years occupied with protective coatings, he has been with Amerocoat Corp. since receiving a bachelor's degree in chemistry from Pomona College in 1940. The bulk of his experience relates to industrial corrosion problems and coatings to solve them.

HENRY T. RUDOLF—President, Atlantic a bachelor's and a professional engineer's degree Coatings Co., Inc., Jacksonville, Fla., he has a bachelor's and a professional engineer's degree from Lafayette College. Until 1958 he was with the steel industry in the United States and abroad. He is author of papers on and holder of patents relating to blast furnace operation. He is active in NACE and was a founder and first chairman of Jacksonville Section.

L. ROBERT WHITING—A member of several NACE technical committees concerned with surface preparation and organic protective coatings, he formerly was chairman of T-6G. With Bakelite Company, Division, Union Carbide Corp. he has worked on polymer chemistry and coatings development. During World War II he worked on government projects in developing anti-corrosive and anti-fouling coatings. He is now with Bakelite's New Products Engineering Dept. A graduate of University of Kansas, 1921, he worked from 1921 to 1924 in the petroleum industry as a chemist, before joining Bakelite.

ABSTRACTS

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graphic examination of scale layers combined with X-ray diffraction and spectrographic analysis is used to determine the effect of alloy composition and moisture in the air.

Effect of Molten Boron Oxide on Selected High Temperature Alloys by David Roller, Wright Air Development Center, Wright-Patterson AFB, Ohio and Charles R. Andrews, University of Dayton.

Industrial and military use of boron and boron-containing compounds has increased greatly in the past two decades. Boron oxide (B_2O_3) is of particular interest. Molten boron oxide, a viscous liquid at 850-3600 F, has been used widely as a solder flux because it can clean metal surfaces by removing oxide layers. Initial studies are reported wherein unstressed high temperature wrought alloys, representing four main composition classes, were exposed to molten boron oxide at test conditions ranging from 1600 to 2000 F in air and inert atmospheres for about 150 hours. A special high temperature furnace operating under reproducible and closely controlled conditions alternately cycled specimens between molten boron oxide and gas environments.

Molten boron oxide proved very corrosive, particularly to iron and cobalt base alloys over 1800 F. Some constituents such as carbon, aluminum, titanium and iron at low concentrations appear to reduce resistance to molten boron oxide. Effects of atmosphere, temperature, alloy composition and other factors are evaluated. Several corrosion mechanisms are postulated to explain data.

How You Can Get Copies Of Conference Papers

Technical papers presented during the 14th Annual Conference at San Francisco will be published in CORROSION as approved by the editors beginning with the April issue. Members of the National Association of Corrosion Engineers will, therefore, ultimately receive copies of all papers approved for publication. Because they are included in CORROSION and are indexed topically and otherwise, the necessity of maintaining separate files and indexes is eliminated.

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NO COPIES OF CONFERENCE PAPERS ARE AVAILABLE FROM CENTRAL OFFICE NACE.

New Marine Corrosion Control Measures Are Covered in Four-Paper Symposium

Realization that real progress can be made in control of corrosion of marine structures has revived interest in new control means and resulted in the preparation of papers such as those being presented during the Marine Corrosion Symposium at San Francisco. Summaries of work done by naval engineers will be made in two papers while a third will cover effect of industrial wastes on corrosion rates. A fourth paper considers electrode properties of mild steel in sea water, always important in cathodic protection design.

May

Wright

ABSTRACTS

Corrosion Problems at Navy Facilities on Tropical Pacific Islands by Kenneth E. Wright, Bureau of Yards and Docks, Dept. of the Navy, Washington.

A coordinated investigation of the corrosion and other deterioration encountered at Navy facilities on tropical Pacific islands has been inaugurated in an endeavor to determine which materials and/or methods of construction will result in the most reasonable initial construction and minimum maintenance costs. High salt content of the marine atmosphere is the basic reason for extreme corrosion and high maintenance costs in certain areas. Variations in salt content result in much more severe corrosion in some locations than in others which climatically are identical or very similar. Relationship between corrosion rates and climate, orientation and geography of the islands is discussed. It is still under study.

Certain metals and alloys have been found to be subject to rapid corrosion and pitting due to the salt laden atmosphere, temperature, and moisture conditions. A list of some materials used with good and bad results is given, as are possible solutions of these and other problems still being investigated.

Marine Structures as Affected by Industrial Wastes by Mark W. Battersby, Seattle, Wash. Common errors in designing cathodic protection for marine structures exposed to industrial wastes are considered. Danger involved in using data derived from observations over short periods instead of year around are considered. A description of cathodic protection methods used on hulls in brackish tidewaters is given.

Problems involved in mitigating corrosion on piers and bulkheads in metropolitan ports are enumerated. Effect of temperatures in Alaska and arctic waters on cathodic protection and corrosion is described.

Comments are made on the effect of marine organisms on metals in far northern waters as contrasted to their effects on the Gulf Coast or in the Mediterranean.

Some Electrode Properties of Mild Steel in Sea Water by T. P. May, Harbor Island Test Station The International Nickel Co., Inc., Harbor Island, N. C.

The potential of freely corroding steel is reported for several conditions of exposure to sea water: namely, quiescent, several velocities and alternate immersion at the tidal zone. The anodic and cathodic behavior of steel in sea water is discussed with reference to galvanic corrosion, cathodic protection and influence of films on the metal surface.

Corrosion Challenges in Naval and Marine Services, by Cdr. F. G. Archbold.

BIOGRAPHIES

MARK W. BATTERSBY—Among other activities in ASME, AACE and NACE Mr. Battersby organized the Puget Sound Section of NACE and served as its first chairman. He has been active in corrosion mitigation work in the Northwest since 1947. He has a BA, economics major, Seattle University and is taking work toward an advanced degree in cost engineering.

THOMAS P. MAY—Since June 1957 Dr. May has been manager of The International Nickel Co., Inc. Kure Beach-Harbor Island Testing Station on the North Carolina coast near Wilmington. He had been technical manager of the station since August 1954. He joined Inco at New York in 1947 after 10 years as head of the Corrosion Section, Chemistry Division, U.S. Naval Research Laboratory, Washington. A director of NACE, he has been chairman of the association's publication committee during the past two years.

KENNETH E. WRIGHT—Assistant to the manager of the Specifications Branch, Bureau of Yards and Docks, Navy. He is on committees developing ASTM, federal and military specifications. He has a BS in civil engineering, University of North Dakota, 1925; was with the state highway departments of North Carolina and Michigan and has been with the Navy Bureau of Yards and Docks since 1931.

BIOGRAPHIES

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ager of its Development and Research Division since June, 1954, Mr. LaQue has spent the better part of his time since graduation from Queen's University, Kingston, Ont., in 1927 doing corrosion research. He is a member of numerous engineering societies concerned with materials, has published numerous papers on corrosion mitigation and is a vigorous proponent of the necessity for long-term service testing to provide reliable corrosion control data. He received the NACE Speller Award in 1949, among other honors from engineering societies, and is sought after as a speaker on corrosion problems.

R. E. MAERSCH—Chief Development Metallurgist, Ampco Metal, Inc., Milwaukee. He received a BS in chemical engineering, University of Wisconsin, 1937, after which he worked for Gisholt Machine Co. He also worked on quality control in the aircraft division of J. I. Case Co., Rockford, Ill. He belongs to ASM, AWS and NSPE.

LOUIS REZNEK—Director of Engineering and Safety, National Tank Truck Carriers, a trade association of for-hire tank truck operators since October, 1953, he has had long experience in motor vehicle operations. He was with the Interstate Commerce Commission 1932-53 as a clerk and engineer, except for army service 1942-45, he is a registered engineer and a member of the bar in the District of Columbia. He is a graduate in mechanical engineering and law, George Washington University, Washington, D. C. His work as chief engineer of the Bureau of Motor Carriers, ICC, put him in charge of equipment and maintenance regulations for motor vehicles included design of tank motor vehicles.

CHARLES R. SOUTHWELL—In materials engineering since 1954 engaged in investigating corrosion and protective coating of metals for Naval Research Laboratory, Canal Zone. From 1950 to 1954 he was engineer-in-charge of concrete and protective coatings during construction of International Falcon Dam and before 1950 worked with Special Engineering Division, Panama Canal Company on research pertaining to corrosion, coatings and concrete for design of a sea level canal. He attended St. Mary's University of Texas, majoring in physics.

G. L. STRAGAND—Assistant Technologist, Applied Research Laboratory, United States Steel Corp., Monroeville, Pa. He has a BS in chemistry, University of Pittsburgh, 1943 and an MS in chemistry from the same school, 1950. His experience includes work in the chemical laboratory of the Carnegie Steel Company and his present work in gas-metal reactions during tin mill box annealing.

PATRICK A. TULLY—Wrought Products research metallurgist for Ampco Metal, Inc., Milwaukee, where he has been employed since graduation in 1951 from University of Wisconsin. He has an MS in metallurgy from Wisconsin, 1955, with a thesis on stress corrosion cracking. He is a member of ASM and an instructor in metallurgy in the evening division of the University of Wisconsin in Milwaukee.

DONALD WARREN—Research project engineer, Engineering Research Laboratory, E. I. duPont de Nemours & Co., Inc., Wilmington, Del. He has a BS in metallurgical engineering, University of Kentucky and MS and PhD degrees from Lehigh University. He joined duPont in 1951 and was an air force pilot during World War II.

ABSTRACTS

(Continued From Page 98)

tors appear of little value in this use and that some in the experimental stage are effective.

Considerable difficulty has been encountered in obtaining consistently reproducible answers by the MIL-I-25017 modification of the ASTM Turbine Oil Corrosion Test. An evaluation technique described by Sinclair has been tried and found to yield inconsistent results. However, the data were somewhat better than those obtained by following the present specification method.

The most hopeful results were obtained by using a corrosion probe of the type described by the Pure Oil Company in place of the corrosion coupon. Families of curves for different concentrations of inhibitor have been obtained with a rather good degree of reproducibility. A clear differentiation has been established between concentrations at which given corrosion inhibitors substantially eliminate corrosion. It is felt this method has considerable merit since it does give a quantitative rating in place of the subjective rating highly susceptible to differences among operators and variations in sample preparation used in previous methods.

Evaluation of Certain Organic Nitrogen Compounds as Corrosion Inhibitors by E. J. Schwoegler, Armour Research Foundation, Chicago.

The static water drop test adapted to research methods has been utilized to evaluate a number of heterocyclic nitrogen compounds. Most of these heterocyclic N derivatives were synthesized and purified and evaluated in a petroleum type lubricating oil and synthetic diester type lubricating oil.

Effect of various functional groups and their positions in the molecule were related to the corrosion inhibiting properties.

Control of Internal Corrosion of Products Pipe Lines With Oil Soluble Inhibitors by M. R. Barusch, California Research Corp.; R. L. Piehl, Standard Oil Co. of Cal.; and L. G. Haskell, Salt Lake Pipeline Co.

Corrosion control in a products pipe line transporting refined petroleum products from Salt Lake City, Utah to Spokane, Washington is discussed. Since this pipeline is a common carrier it handles such stocks as motor Diesel and aircraft fuels and alkylate. Some fuels carry different corrosion inhibitors while many others are without any inhibitor. A theory is proposed to explain the mode of action of corrosion inhibiting when a variety of different inhibitors are used in the same pipe line.

BIOGRAPHIES

R. B. THOMPSON—Since 1939 in the research and development laboratories of Universal Oil Products Company, he has been working with additives, isomerization and alkylation of paraffin hydrocarbons. He has been a degree in education from Southern Illinois University, Carbondale, 1934 and a PhD, 1938, Harvard where he worked on the university staff for a year with James B. Conant.

CHARLES WANKAT—He has been employed at Universal Oil Products Company research laboratories since 1936. He has a BSc in chemistry, Illinois Institute of Technology, 1942. His principal work is analytical chemistry related to treating petroleum distillates, refinery service and corrosion studies. He is now supervisor of the UOP service laboratory at Des Plaines, Ill.

Bilhartz

Eldredge

Fraser

Greenwell

Kerr

Prange

Mechanical Properties of Oil Well Tubing Among Oil and Gas Symposium Topics

The subject of protection of oil and gas well equipment always has high interest among persons attending NACE Conferences and the quality of the papers in the Oil and Gas Production Symposium will make San Francisco's meeting no exception.

Of the seven papers scheduled, two consider the mechanical properties of and effect of dissolved gases on tubing; another reviews the gas condensate well corrosion problem and a fourth discusses cathodic protection of 480 oil well casings.

Other papers pertain to sulfide corrosion cracking and water flood operations.

Treseder

Watkins

ABSTRACTS

Corrosion of Steel in Water by Varied Ratios of Dissolved Gases, by J. Wade Watkins and G. W. Kincheloe, Bureau of Mines, U. S. Dept. of Interior, Bartlesville, Okla.

A basic study of the corrosivity to steel in water of varied concentrations of the dissolved gases, oxygen, free carbon dioxide, and hydrogen sulfide was reported in an earlier paper. Additional laboratory tests were made with varied ratios of free carbon dioxide to both oxygen and hydrogen sulfide, with the concentration of one gas maintained constant throughout a series of tests and the concentration of the other gas varied by increments to values approaching saturation. Corrosion rate as a function of time in the system was determined at time intervals to a maximum of 984 hours' exposure. Results indicated that corrosion caused by oxygen and free carbon dioxide increased almost in direct proportion to the concentration of dissolved oxygen, the greatest potential corrosion in a water-injection system might be expected from water saturated with air and containing high concentrations of dissolved free carbon dioxide and the inhibiting action of hydrogen sulfide by the formation of protective coatings was manifested almost throughout the entire range of carbon dioxide-hydrogen sulfide ratios. In the time versus corrosion tests, the corrosion rate was fairly linear with time up to about 72 hours, increased, and again assumed a nearly linear relationship from about 140 hours to 984 hours' exposure.

Mechanical Properties and Corrosion Resistance of Oil Well Tubing by F. A. Prange, Phillips Petroleum Co., Bartlesville, Okla.

A resume of the applications of various grades of tubing in sweet and sour condensate wells and sweet and sour oil wells. Modes of corrosion damage in the different tubing materials are discussed. Sour condensate wells are particularly troublesome since sulfide stress cracking is closely related to the mechanical properties. Stress corrosion cracking may be a serious problem in the high strength materials. Mechanical properties and the effects on service are examined. Transverse notch ductility is a critical factor in high strength steels. Common defects found in the various grades are discussed.

A Review of the Gas Condensate Well Corrosion Problem by D. R. Fincher, Tidewater Oil Co., Houston, Texas.

Emphasis is placed on "super" high pressure condensate wells with shut-in pressure over 5000 psi. Present status of corrosion control in gas condensate wells is given in comparison with data reported in the 1953 book, "Condensate Well Corrosion," of the Natural Gasoline Association of America. An estimate of the current value of the NGAA book is given.

Cathodic Protection of 480 Deep Oil Well Casings by A. W. Kerr, Shell Oil Co., Ventura, Cal.

Cathodic protection has been installed on some 480 oil wells in the Ventura Field by the Shell Oil Company to alleviate external casing corrosion which has been a problem for many years. Extensive field investigations and laboratory studies were made to evaluate various factors associated with cathodic protection. Current requirements were based upon down hole current-potential curves utilizing a new interpretation. An economic analysis of the test data illustrated that an installation producing 27 amperes per well would give maximum profits. Depth of protection was shown to be adequate to 9000 feet and the surface pipe effect was found to be of no major consequence.

An installation of 27 wells for pilot testing was completed in March 1956 with the major installation being completed in January 1957. Although it is too early to evaluate absolute effectiveness,

(Continued on Page 102)

BIOGRAPHIES

H. L. BILHARTZ—President of Production Profits, Inc., a firm of consulting engineers in the petroleum production field. He has been a region and section officer and has arranged programs for local and national meetings. His activity on NACE technical committees includes chairmanship of T-1 and membership in several others in the oil production field. He also is a member of Natural Gasoline Association of America, API and AIME. He graduated from Southwestern University and attended graduate schools at Harvard and Massachusetts Institute of Technology.

G. G. ELDREDGE—A member of the Statistics and Operation Research Group, Applied Mathematics Dept., Shell Development Co. He has worked on corrosion research since 1946 and was employed similarly at Aluminum Research Laboratories, New Kensington, Pa. He has a PhD, 1940, University of Minnesota and is author of papers on inhibitors, statistical analysis of corrosion pitting and others.

(Continued on Page 102)

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ABSTRACTS

(Continued From Page 101)

the Ventura tests indicate that cathodic protection is feasible and will be highly profitable for deep oil wells.

Laboratory and Field Methods for Quantitative Study of Sulfide Corrosion Cracking by J. P. Fraser, G. G. Eldredge and R. S. Treseder, Shell Development Co., Emeryville, Cal.

Quantitative procedures for sulfide corrosion cracking tests, both in the laboratory and in the field, have been developed. Statistical technique of probit analysis has been adapted for use with cracking test data so as to make optimum use of a minimum number of tests.

Analysis of data from laboratory tests yields a number which is called the critical stress, S_c. This is the stress at which the probability of failure under the specific test conditions used is one-half. Very susceptible steels have low S_c values whereas non-susceptible steels have high S_c values. The critical stress is a function not only of the alloy tested but also of the test environment and procedure. It is lowered by addition of acids (especially organic acids such as acetic) and carbon dioxide.

Using alloys of varying S_c values, all tested at the same high stress, the relative severity, R_s, of any given test environment (i.e., flow from a sour gas well) can be measured. Analysis of the data from field tests yields a number, R_{sc}, which is the critical stress of an alloy which would be expected to give 50 percent failures in test. High R_s values are associated with very severe test environments and low R_s values with mild environments.

This general method may be adaptable to other stress corrosion systems and thus provide a quantitative means of expressing susceptibility to attack or severity of an environment.

Influence of Metallurgical Variables on Resistance of Steels to Sulfide Corrosion Cracking by J. P. Fraser and G. G. Eldredge, Shell Development Co., Emeryville, Cal.

A statistical study has been made of sulfide corrosion cracking of steel as a function of chemical composition, mechanical properties and heat treatment. Of 104 different alloys studied 79 were commercially-produced API grades J-55, N-80 and P-110 steels. It was possible to derive predictive equations which can be used to rate the cracking susceptibility of a new alloy from its known metallurgical properties. Properties that most strongly influence resistance to corrosion cracking are reported.

An additional result of this investigation is the demonstration that resistance to cracking can be markedly improved by tempering N-80 steel for a half hour at 1100 F. The accompanying loss of yield strength is not excessive.

Corrosion in Water Flood Operations by H. E. Greenwell and H. L. Bilhartz, Production Profits, Inc., Dallas, Texas.

BIOGRAPHIES

(Continued From Page 101)

J. P. FRASER—Chemist, Materials Engineering and Corrosion Dept., Shell Development Co., Emeryville, Cal., he graduated from Cornell in 1947 with a BChemE and received a PhD in metallurgy from the same school in 1949. His work with Shell since 1949 has been in materials engineering and corrosion research. A member of NACE, he is active in technical committee 1-F. He is author of several papers on his specialties.

H. E. GREENWELL—Now with Production Profits, Inc., Dallas, he has been doing corrosion control work on oil wells for more than 8 years, principally with Atlantic Refining Co. He is a member of the Natural Gasoline Association of America, and is active on NACE technical committees dealing with oil well corrosion. His published work includes studies on corrosion characteristics of oil wells and methods of controlling corrosion damage in production equipment.

A. W. KERR—A member of the mechanical engineering staff in the Production Dept., Shell Oil Co., Ventura, Cal. working on oil well casing corrosion problems and drilling and production matters. He has a BS in mechanical engineering, 1952, Oregon State College and joined Shell on graduation.

F. A. PRANGE—His work in the engineering department of Phillips Petroleum Company, Bartlesville, Okla. Since 1941 has been on corrosion and metallurgical problems in the production and refining of oil and manufacture of petrochemicals. He has a BS in metallurgical engineering, Purdue, 1940 and an MS in 1941.

R. S. TRESEDER—Development Supervisor, Materials Engineering and Corrosion Dept., Shell Development Co., Emeryville, Cal. A 1939 graduate of University of Utah with a BS in chemical engineering, he was employed by Shell the same year. Since 1940 his work has concerned research and development on a wide variety of corrosion problems associated with petroleum and chemical industries. He is author and co-author of numerous technical papers, a member of several NACE technical committees, past chairman of the San Francisco Bay Area Section and Western Region.

Please Turn to PAGE 75 for TENTATIVE TECHNICAL COMMITTEE SCHEDULE and Summaries of Probable Agendas

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Pipeline Symposium Will Cover Coatings, Cathodic Protection and Potential Data

Papers on coatings and their evaluation, cathodic protection in high resistivity soil and a discussion of the theory of potentials of underground structures will be given during the Pipe Line Symposium at San Francisco.

Authors represent two large pipeline companies, a leading manufacturer of coatings and a consulting engineer of long experience.

Allen

Scott

Shideler

Van Nouhuys

Whittier

BIOGRAPHIES

E. R. ALLEN, JR.—Corrosion engineer, Humble Pipe Line Co. since 1949, testing and evaluating external pipe line coatings, internal pipe liners and sour crude corrosion inhibitors. In 1954-56 as an Air Force Project Officer he worked on aircraft corrosion from rocket blast residues. Has a BS, chemical engineering, 1948, Massachusetts Institute of Technology and BS, 1949 in general engineering.

GORDON N. SCOTT—Since 1927 Mr. Scott has been engaged in research and consulting work connected with pipe line corrosion. He received

(Continued on Page 104)

ABSTRACTS

Thin Film Coatings for Protection of Underground Pipe Lines by N. T. Shideler and F. C. Whittier, Pittsburgh Coke & Chemical Co., Pittsburgh.

The pipe line industry is constantly searching for new and better coatings to protect underground pipe lines against corrosion. Coatings applied hot in thickness of approximately 3/32-inch have been predominant for a great many years. Because they are hot applied and they require considerable application equipment and are subject to soil stress and mechanical damage during installation, an effort is being made to develop new cold applied coatings suitable for underground pipe using relatively thin films in the range of 5 to 30 mils thickness.

Two types of cold applied coatings are being tested, both based on epoxy resins cured by catalytic action, rather than by solvent evaporation. Epoxy resins used usually have a molecular weight range of 350 to 700. The resins are catalytically set by amine type catalysts. One product is made from straight epoxy resin, solvents and mineral pigments and is applied in two coats in a thickness range of 5 to 8 mils. The second type of coating is a combination of the epoxy resins with coal tar or coal tar pitch, solvent and mineral pigments using the same type catalyst as the first product. The latter product can be applied in thicknesses ranging from 15 to 30 mils in either one or two coats. These coatings have been on test for approximately four years on joints and small sections of pipe. However, moderate lengths of small diameter pipe lines have been on test for approximately two years. Characteristics of both coatings include excellent adhesion and resistance to mineral acids and alkalies and to petroleum products. Both are extremely hard and tough and they are not affected by soil stress and not easily mechanically damaged. Test results thus far are excellent. The only disadvantages are the time required for polymerization of the resin and some application difficulties.

(Continued on Page 104)

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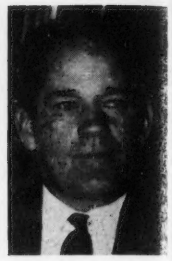

Severance

Use of Plastic as Engineering Material Is Principal Theme of 4-Paper Symposium

Application of plastics as engineering material grows as fast as reliable data on performance can be accumulated. In the four-paper Plastics Symposium at San Francisco, subjects to be covered are application of polyvinylchloride pipe, reinforced plastic structures, creep studies and application of reinforced epoxy pipe.

One paper considers the influence exerted on the engineering use of polyvinylchloride by efforts of NACE technical committees.

ABSTRACTS

Practical Applications and Recommendations for the Use of Polyvinylchloride Sheets and Piping —The NACE Influence by Louis Perret, Jos. T. Ryerson & Sons, Inc., Chicago.

Use of polyvinly chloride materials in the United States and Europe is reviewed, with an explanation of how they help in solving corrosion problems and their limitation.

Technical education for fabricators and manufacturers is described.

Application of polyvinyl chloride sheets is described, with suggestions on the choice of materials and welding systems for a variety of corrosive environments. Correct and incorrect ways to install polyvinyl chloride pipe are defined. Data are given on expansion in PVC pipelines.

The influence of work done by the National Association of Corrosion Engineers on PVC applications is reported.

Reinforced Plastic Structures for Corrosion Control by W. A. Severance, Ceilcote Co., Cleveland.

Test results are shown which illustrate the versatility of reinforced plastics in solving corrosion problems. Special study has been made of resistance to solvents, organic acids and concentrated inorganic acids, which may be termed "fringe" applications for plastics. Objective is to illustrate differences among various polyesters and epoxies in specific chemicals.

A series of tests in wet chlorine at 210 F shows wide variations in the resistance of different brands of polyesters. Results of tests are shown graphically. They indicate that compounds showing best resistance should be worthy of extensive field testing. Physical test values are given for laminates used in chemical service. Laminates should be designed for maximum chemical resistance; this does not always parallel maximum strength. Factors used in engineering reinforced plastic structures are also given.

Numerous photos show the variety of equipment that can be fabricated of reinforced plastics.

Three-Year Creep Studies on Polyvinyl Chloride Pipe and Fittings, G. H. Hunt and H. H. George, Tube Turns Plastics, Inc., Louisville.

Application File on Reinforced Epoxy Pipe by Edward D. Edmisten and H. D. Boggs, Fibercast Corp., Sand Springs, Okla.

ABSTRACTS

(Continued From Page 103)

In general, cost of these coatings applied cold to pipe is the same or only slightly higher than the cost of the hot applied coatings.

Cathodic Protection and High Resistivity Soil—A Sequel by H. C. Van Nouhuys, Southeastern Pipe Line Co., Atlanta, Ga.

At the 1953 NACE Conference a paper entitled "Cathodic Protection and High Resistivity Soil," described the installation of a rectifier system designed on the basis of the remote electrode criterion. This 25-rectifier system represented an attempt to obtain solid protection of 350 miles of bare 8-inch products pipe line in the ultra-high resistivity soil of Georgia using current densities of unusually low order.

Five years and eight leaks later this sequel is written.

Analysis of the leak record is made from data obtained at each leak location in an effort to properly locate those leaks due to inadequacy of the criterion as opposed to leaks resulting from rectifier down-time or other unusual causes.

A summary is given of the savings to be expected from use of this method of design over other criteria as well as precautions to be taken in its use.

A Coating Evaluation Testing Program by E. R. Allen, Jr., Humble Pipe Line Company, Houston.

Factors to be considered in establishing a testing program for the evaluation of pipe line coatings are outlined. Testing procedures are described and the results obtained are discussed. New developments in coatings require modification of some testing criteria.

Base Potentials of Underground Structures by Gordon N. Scott, Los Angeles, Cal.

BIOGRAPHIES

(Continued From Page 103)

the NACE Speller Award in 1955, has a PhD in physical chemistry and other degrees from University of California. He is author of numerous papers on underground corrosion and cathodic protection.

N. T. SHIDELER—Since 1930 he has been employed by various companies producing coal tar and, in general, coal tar coatings. He was in the research laboratories of Reilly Tar & Chemical Corp. for six years and later was associated with Wailes-Dove-Hermiston (later Koppers Company) until 1948 when he joined Pittsburgh Coke & Chemical Company. He started their production of coal tar coatings and for the past several years has been handling development, production and sales of coal tar coatings exclusively. He recently was appointed president of Insul-Mastic, Inc., a subsidiary of Pittsburgh Coke & Chemical and directs its research activities as well as those of Pittsburgh Coke.

HERBERT C. VAN NOUHUYS—Corrosion engineer, Southeastern Pipe Line Company. Since 1934 he has been engaged in various engineering phases of pipelining. He has a BS, 1934, electrical engineering, University of Michigan. During World War II, he was with the Signal and Air Corps as a training center staff officer, and technical officer at radar research and development laboratories. He has been a regional officer and presently is a member of the NACE board of directors.

BIOGRAPHIES

H. D. BOGGS—General manager, Fibercast Corp., Sand Springs, Okla. He was with Glen L. Martin Aircraft Co., resigning as assistant to the vice-president and general manager; was production manager for two years with Goslin Birmingham Mfg. Co. and is a graduate of Columbia Tech in aeronautical engineering. He has been interested in reinforced plastic products and their development since 1947.

EDWARD D. EDMISTEN—Quality control manager, Fibercast Corp., Sand Springs, Okla., he has degrees in chemistry and petroleum engineering from University of Tulsa and Oklahoma Baptist University. For 15 years he was in the field and research centers of Pan-American Petroleum Co. He is a member of AIME, ASM, SPI and ASTM.

HENRY H. GEORGE—Manager of research and chief products engineer, Tube Turns, a division of National Cylinder Gas Co. He has been with Tube Turns since 1944, and had previously been employed in various capacities by Jones and Laughlin Steel Corp., Pittsburgh Water Heater Co., Wm. B. Scaife Co. and with Technicolor as a motion picture engineer. He has a BS, physics and engineering, 1935, University of Pittsburgh; MS, physics, 1939 and worked toward a PhD until 1942. He is a member of American Institute of Physics, Society for Experimental Stress Analysis, American Society for Metals and Kentucky Society for Professional Engineers.

GEORGE H. HUNT, III—Manager of research and quality control for Tube Turns Plastics, Inc. since June, 1957, he was a research and development engineer for his employer for a year before taking his present position. He has a BA in physics, University of Louisville, and is a member of American Society for Quality Control and Sigma Phi Sigma, physics honor society.

LOUIS R. PERRET—Since 1955, plastics engineer and technical adviser in Joseph T. Ryerson & Sons, Chicago. A native of Italy, he graduated from Italian technical schools, was in the Italian army 1931-32. After two years of training in the chemical construction engineering office, Paris, he became plant superintendent in coal and by-products distillation in Marquise, France; for two years he was superintendent in glycerin-fatty acids and sulfonation plant in Borgars, Italy and during 1945-55 was plant manager and sales manager in a polyvinyl chloride extrusion and fabrication plant in Turin.

W. A. SEVERANCE—Chief chemist, The Ceilcote Co., Inc. in charge of research, development and technical service for corrosion resistant materials of construction. He has a BS, 1939, in chemistry, University of Wisconsin. He formerly was employed as materials control chemist, Maytag Company, 1939-42; chemist in research and development, Parker Appliance Co. He joined Ceilcote in 1949.

F. C. WHITTIER—Supervisor of protective coating research and development at Pittsburgh Coke and Chemical Co. He has been employed by United States Testing Laboratories, and from 1935 to 1946 was employed by Barrett Division, Allied Chemical & Dye Corp. as research chemist, technical representative on coatings and chief plant chemist. He graduated from Bowdoin College in 1933. Almost all of his work since graduation has been on coatings.

Ultimate Reasons for Reactions Considered In Three Papers

Investigation into the ultimate reasons for reactions is important because they illustrate the principles that apply to materials deterioration. Three papers in the Corrosion Principles Symposium consider some of the fundamental aspects of corrosion processes.

Electron microscope studies of single metal crystals are covered in one paper in which the author describes the reaction of single large crystals under corrosive conditions. Another paper analyzes literature on pitting corrosion for the last 35 years and a third paper discusses surface area relationships in polarization and corrosion.

ABSTRACTS

Corrosion of Metal Single Crystals as Seen With the Electron Microscope by Kenneth R. Lawless, Dept. of Chemistry, University of Virginia.

The importance of looking at a metal surface, both before and after corrosion, should be obvious. All too frequently, however, measurements on corrosion are reported in which no information is available on the structure before and after the reaction. This paper will report some observations on the structure of surfaces of copper single crystals and large grain crystals with grain and sub-grain boundaries, before and after corrosion in air and in copper sulfate solution, particularly as observed with the electron microscope.

Use of single crystals makes possible the study of the effects of crystal face and of imperfections within the crystal. Large grained samples have been used in studying the effects of grain and sub-grain boundaries. Utilization of the high resolving power and depth of field of the electron microscope makes possible the observation in deail of many phenomena such as etching and oxide growth which could not be satisfactorily studied by other techniques.

The amount and type of oxidations varied markedly with crystal face. Under oxidizing conditions, three types of structure occurred: 1. A thin polycrystalline oxide next to the metal. 2. Small oriented single crystal oxide nuclei, and 3. Large oriented single crystal polyhedra with well formed shapes. In the case of oxidation in solution, spirals were seen on the larger oxide figures. The effect of grain boundaries was very complex, results depending on the angle of tilt between the crystals and on their specific orientation. In some cases very large oxide polyhedra were observed on a grain boundary, in other cases rows of polyhedra were parallel to a boundary, but not on it, and in other cases oxide polyhedra did not form at a boundary.

The type and amount of etching varied with crystal face, the pH of the etching solution, the history of the sample, i.e. whether strained, or whether annealed in hydrogen to remove any oxide film and on the presence of imperfections in the crystal.

Some examples of "catalytic corrosion" of a metal surface also are shown.

A Critical Analysis of Pitting Corrosion by M. G. Fontana and N. D. Greene, Dept. of Metallurgical Eng., Ohio State University, Columbus.

A critical analysis of the literature on pitting corrosion published during the past 35 years. The definition of pitting, the occurrence of pitting and the factors influencing this type of attack are considered. Electrochemical, morphological and physicochemical aspects are discussed. An examination of the theories of pitting and the methods used to study this form of corrosion are also presented.

Surface Area Relationships in Polarization and Corrosion by Milton Stern, Metals Res. Lab., Electro Metallurgical Co., Div. Union Carbide Corp.

Polarization behavior of surfaces containing regions which exhibit different overvoltage parameters for the same reaction are described. Also, the effect of cathode area fraction on corrosion potential and corrosion rate is considered for a corroding surface controlled by activation polarization. It is shown that the corrosion potential and corrosion rate are most sensitive to area changes when the surface is largely cathode or anode. The corrosion current reaches a maximum when the surface is equally divided into anodic and cathodic regions.

Fontana

Lawless

Stern

BIOGRAPHIES

MARS G. FONTANA—Professor of metallurgical engineering and research for Ohio State University, he is a past president of NACE. He holds several degrees and has received numerous honors from engineering societies for his work on corrosion control. An author and co-author of techni-

(Continued on Page 106)

Corrosion Can Take a Holiday

IN PIPE COATINGS AND WRAPPINGS OR IN PROTECTIVE PAINTS

Tinker and Rasor Holiday Detectors can assure this fact. They are an insurance policy that you are getting what you pay for in applied protective coatings. Tinker and Rasor pipeline (jeeps) holiday detectors are your inspector's right hand. They can give him the best quality control possible. On transmission lines he can spot trouble, as soon as it develops, such as clogged sprays on dope machine (which leaves pipe uncoated but covered with outerwrap), carbonized particles in bitumens, etc.

MODEL HP US Patent #2629002 (electrode)

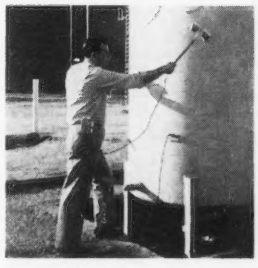
LINE-TRAVELING

The model HP self-contained line-traveling detector, has its own power supply (wet cell battery); needs no ground wire and operates equally well on damp or dry pipe. Adaptable on any size transmission pipelines.

SHOULDER-PACK

Tinker and Rasor shoulder-pack holiday detector is designed to give the best possible inspection. It offers an adjustable voltage output range from 5,000 to 20,000 volts of sharp pulse type voltage which puts less stress on protective coatings. This equipment is available with conductive silicone brush type electrodes (non-fouling in soft coatings), for inspecting flat or irregular surfaces and with full and a half circle spring electrodes. This model will inspect damp or dry coating equally well and, as in all Tinker and Rasor detectors, has audible bell signal.

MODEL EP



MODEL M 1

PAINT AND THIN FILM

Tinker and Rasor Model M 1 detector is used for inspection of thin films where complete protection is a necessity. The detector has a bell alarm signal and is equipped with a leather case and belt loop. The potential of inspection is 67 volts. Not a spark detector, it operates with a damp cellulose sponge mounted on a phenolic wand handle. Pin holes can be pin pointed by using a corner of the sponge once a holiday has been detected.

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Pulp, Paper Corrosion Topic of Three Papers

Baisch

Lundberg

Three papers dealing with the problems of pulp and paper manufacturers are expected to attract wide attention among engineers in this industry in the Northwest and in Western Canada. The papers cover corrosion activities of the Technical Association of the Pulp and Paper Industry, corrosion associated with "wet strength" solutions and those encountered in sulfur burning and acid making.

ABSTRACTS

Review of Recent Corrosion Activities in the Technical Association of the Pulp and Paper Industry by Steve Basich, Thilmany Pulp and Paper Co., Kaukauna, Wis.

A Digester Corrosion Sub-Committee was formed in February 1950 under the chemical engineering committee of the Technical Association of the Pulp and Paper Industry immediately after the Kraft pulping section reported a severe corrosion problem in digesters.

After five years of investigation and publication of much data, the subcommittee was expanded to a full committee so other major corrosion problems in the industry could be considered.

Some of the activities and scope of planned activities in combatting corrosion in pulp and paper making are reviewed.

Materials, Behavior and Corrosion Characteristics in Paper Mill "Wet Strength" Solutions by I. S. Levinson, Ampco Metal, Inc., Milwaukee.

Some corrosion and its effect on volume and quality of product resulting from acidified solutions containing certain melamine resins used to impart "wet strength" to papers is reviewed. Testing programs instituted as a result of this corrosion and aluminum bronze equipment designed to replace other materials suffering corrosion damage are described.

BIOGRAPHIES

STEPHEN J. BAISCH—Chief design engineer, Thilmany Pulp and Paper Co., Kaukauna, Wis., he has a BS, 1942, in mechanical engineering, University of Wisconsin. For three and a half years during World War II he was an officer in the Army Ordnance Department. He is chairman of the Technical Institute of the Pulp and Paper Industry Corrosion Committee, past chairman of the Lake State Tappi Section and a delegate from Tappi to the NACE Inter Society Corrosion Committee.

A. H. LUNDBERG—A member of the firm of A. H. Lundberg, Inc., Seattle, Wash. Born and educated in Sweden, he was employed in Canada in 1921 as chief chemist, Ha Ha Bay Paper Company, now Consolidated Paper Co., Port Alfred, P. Q. and Fraser Companies, Ltd., Edmundson, N. B. until 1924 when he joined J. D. Jenssen Co., New York as project engineer. In 1927 he moved to Seattle as Western manager for Jenssen and in 1932 started his own business.

Dravnieks

Freedman

Schillmoller

Useful Data on Refinery, Petrochemical Corrosion to Be Presented in Symposium

The active segment of members in NACE interested in controlling corrosion of oil refineries and petrochemical plants will find considerable useful information in the four papers scheduled for the Refinery Industry Symposium at San Francisco. Data on inhibitors will be given in one paper by authors who frequently contribute information in this field.

Three other papers cover tubular products for elevated temperatures, sour water strippers and tank ships.

ABSTRACTS

Evaluation of Refinery Corrosion Inhibitors by A. J. Freedman and A. Dravnieks, Standard Oil Company of Indiana, Whiting, Ind.

Since reproduction of refinery operating conditions in the laboratory is difficult, most laboratory testing of refinery corrosion inhibitors has been unreliable. For this reason, inhibitors for refinery use have been selected mainly on the basis of qualitative field trials. Such tests are expensive and subject to many independent, noncontrollable variables.

Frequently, failure of corrosion inhibitors to provide satisfactory protection can be traced to specific environmental factors. Therefore, new laboratory tests for commercial refinery corrosion inhibitors have been developed to simulate closely certain features of actual field conditions. Inhibitors have been evaluated for corrosion protection in light naphtha condensers, distillation towers, special high temperature locations, high velocity service, narrow crevices, and for impingement attack.

These test data show conclusively that no single corrosion inhibitor can be expected to provide satisfactory corrosion protection in all refinery locations. Plant results obtained with electrical resistance corrosion probes corroborate the laboratory evaluations.

Some Corrosion Considerations in Selection of Tubular Products for Elevated Temperature Service by Thomas Krebs.

Corrosion in Sour Water Strippers by J. F. Mason, Jr., and C. M. Schillmoller, The International Nickel Co., Inc.

Corrosion Prevention in Tankers and Storage Tanks by Fogging or Flotation with an Inhibitor Solution by J. C. D. Oosterhout, M. E. Stanley and W. S. Quimby, The Texas Company, Port Arthur, Texas.

BIOGRAPHIES

ANDREW DRAVNIEKS—Head of Corrosion Section, Material Div., Engineering Research Dept., Standard Oil Company (Indiana). Attended University of Latvia and Marburg University, Germany and received a PhD, 1949, Illinois Institute of Technology. His work includes paint technology, polarography surface chemistry and the kinetics of corrosion processes.

A. J. FREEDMAN—Project engineer, Corrosion Section, Materials Div., Engineering Research Dept., Standard Oil Company (Indiana) Whiting. He has a PhD, 1948, New York University and before joining Standard worked at Massachusetts Institute of Technology, Los Alamos Scientific Laboratory and University of New Mexico.

CHARLES M. SCHILLMOLLER—Now with The International Nickel Co., Inc., Los Angeles, he formerly was a corrosion engineer for Richfield Oil Corp. where he worked on refinery problems. He was educated in Holland, received a degree in chemical engineering from University of Sydney, Australia and was a pilot in the Netherlands Air Force from 1941 to 1947.

BIOGRAPHIES

(Continued From Page 105)

cal articles on corrosion process and control and test procedures, he is a consultant on corrosion problems for several large industrial firms.

KENNETH R. LAWLESS—Since 1952 Research Associate, Department of Chemistry, University of Virginia, he was a Fullbright Fellow, Metallurgical Institute, Norwegian Technical High School, Trondheim, 1951-52. He has a BS, 1946, in chemistry, Lynchburg College; certificate in meteorology, University of California at Los Angeles, 1943 and PhD, 1951, in chemistry, University of Virginia. His field of specialization includes single crystal chemistry, with emphasis on structure of surface and chemical products formed on them; x-ray, electron diffraction and electron microscope techniques.

MILTON STERN—A research metallurgist, Metals Research Laboratories, Electro Metallurgical Company, Union Carbide Corp., he has conducted fundamental research studies on electrode potential behavior, corrosion inhibitors, cathodic protection intergranular corrosion, attack by chlorinated solvents and high temperature oxidation. He has a ScD in physical metallurgy from Massachusetts Institute of Technology, 1952 and was the Weirton Post-Doctoral Fellow from 1952 to 1954. He received the Electrochemical Society's Young Author Award in 1955 and is a member of several engineering societies, including NACE.

Refinery Round Table Discussion

The second half of the Refinery Industry Symposium will be devoted to a round table discussion of refinery industry corrosion problems. R. W. Manuel, Socony Mobil Oil Company will act as moderator and officers of NACE Group Committee T-8 on Refining Industry Corrosion will act as a panel. Officers of T-8 are M. E. Holmberg, consultant, Chairman; E. B. Backensto, Socony Mobil Oil Company, vice-chairman; Cecil Phillips, Humble Oil and Refining Company, secretary.

Members of T-8 will be asked to submit topics for discussion to the round table.

Brindley

Bruckner

Lichtenberger

Scharfstein

Lead Sheathed Cables, Stainless Steels Are Topics for Utilities Symposium Papers

Two papers discuss the corrosion problems associated with lead sheathed cables underground during the Utilities Industry Symposium at San Francisco. The other two papers in the symposium relate to stress corrosion cracking of stainless steels in chlorides and effect of seawater on stainless steel traveling screens.

One of the cable papers considers the economically important problem of protecting lead sheaths exposed to de-icing salts.

ABSTRACTS

A Discussion of Measures Used to Correct Differential Aeration Corrosion of Partially Submerged Lead Sheathed Telephone Cable by David T. Jones.

Telephone lead sheathed cables in congested metropolitan areas generally are installed in a multi-duct structure with manholes spaced approximately 500 to 600 feet apart. The conduit structure usually is graded toward the manholes for drainage. However, foreign substructures sometimes necessitates dips in the conduit structure.

If these dips are not drained, water is entrapped. This water entrapment is discussed with reference to lead sheathed telephone cable corrosion resulting from differential aeration. Solution of the problem by various means is discussed in case histories.

Cathodic protection was tried and the problems involved in obtaining sufficient protective current without causing interference to foreign substructures is discussed also.

Cathodic Protection of Lead Cable Sheath in the Presence of De-Icing Salts by Walter H. Bruckner and W. Wayne Lichtenberger, University of Illinois, Urbana.

Data obtained in the cathodic protection laboratory in the Electrical Engineering Department of the University of Illinois are presented. A previous paper on this subject in CORROSION for February 1957 showed that complete protection of sheath could be effected in a constant current system even in the presence of high salt content and pH. Relationship of protective potentials to pH and salt concentration now has been established for commercial lead sheath alloy composition and is shown to be substantially the same as given in the Pourbaix diagram for pure lead.

The use of a potential gradient for lead cathodes was developed to provide simulated duct tests. Data from gradient tests gave information on effects of salt concentration, pH and potential on corrosion behavior of lead when fully immersed and partially immersed. Criteria for effective cathodic protection are shown to be different for portions of the lead cathode below, at, and above the water line. Most severe corrosion conditions at the air-solution interface require a more negative voltage for protection than for areas below the interface. Distribution of potential along the gradient as a function of salt content, type of salt and current input to the cell shows some important aspects to be considered in the field as possible regions of corrosion hazard.

Chloride Stress Corrosion Cracking of Austenitic Stainless Steel—Effect of Temperature and pH by Lawrence R. Scharfstein and William F. Brindley, Westinghouse Bettis Atomic Power Div., Pittsburgh, Pa.

Overstressed U-bends of Types 304 and 347 stainless steels were exposed to water containing 5 to 550 ppm chloride ion at temperatures of 165, 185, 200, and 500 F. The pH was controlled at 6.5-7.5 and 10.6-11.2. Examination of specimens was made to ascertain the initiation of cracks. At high pH, cracks appeared at the edges with little evidence of piting; at the neutral pH, cracks were found both at edges and associated with pits. Oxygen content of the water was an important variable, especially in its effect in accelerating pitting of stainless steel. Sensitized Type 304 stainless steel had longer and deeper cracks than annealed Types 304 and 347 in the same exposure time. Where boiling or alternate wetting and drying of the stainless steel U-bends did not occur, chloride stress corrosion cracking of the U-bends was prevented by alkaline phosphate treatment.

Corrosion Experience With All-Stainless Steel Traveling Screens in Seawater by William M. Jakway.

BIOGRAPHIES

WILLIAM F. BRINDLEY—Materials engineer, Westinghouse Bettis Atomic Power Division. He has a BS, University of Pittsburgh in 1949. He has been engaged in evaluation of materials for and development of procedures for nuclear powered components.

WALTER H. BRUCKNER—Research associate professor of electrical and metallurgical engineering at University of Illinois. He also is technical director of the university's cathodic protection laboratory. Before coming to the university in 1938 he was on research staffs at Naval Research Laboratory, American Smelting & Refining Co. and Crucible Steel Co. His work has been in physical metallurgy, fatigue, welding, brittle fracture and corrosion. He is a member of several engineering organizations including NACE.

DAVID T. JONES—Supervising Construction Foreman with Pacific Telephone and Telegraph Co., Los Angeles. He graduated from California Institute of Technology in 1925 with a BS in electrical engineering. His work includes outside plant engineering, construction and maintenance and preparation of plans and specifications for telephone plant cable and wire installations. He has been in charge of transmission tests on trunk and toll cables for the Southern Area of his company for 25 years. Past chairman of Western Region and Los Angeles Section NACE, he is a registered professional engineer in California.

W. W. LICHTENBERGER—Research Associate, Controls System Laboratory, University of Illinois, he formerly was employed at the university's cathodic protection laboratory. He has a BS in electrical engineering, 1955, University of Illinois and an MS in 1956. He designed and built much of the equipment in the cathodic protection laboratory and has specialized in electronics and circuits. He has received several honors in recognition of his work at the university.

L. R. SCHARFSTEIN—Senior engineer, Westinghouse Bettis Atomic Power Division, Pittsburgh, he evaluates corrosion resistance of materials in high purity water and related systems. He has a BS, 1946, chemical engineering, Pennsylvania State University, and a PhD, 1953, physical chemistry, New York University.

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TECHNICAL PROGRAM

Chronological Schedule

Following is a chronological summary of most of the technical program arranged for the March 17-21 Fourteenth Annual Conference and Exhibition at San Francisco:

Monday, March 17

Afternoon: Symposia—Pipe line, Protective Coatings, Part 1; Refinery Industry, Part 1.

Tuesday, March 18

Morning: Symposia—Protective Coatings, Part 2; Oil and Gas Production, Part 1; Chemical Industry, Part 1.

Wednesday, March 19

Mornings: Symposium—Corrosion Principles, Refinery Industry, Part 2; Underground Corrosion Coordinating Round Table and T-7 meeting; Corrosion Inhibitor.

Afternoon: Symposia—Cathodic Protection, General Corrosion, Part 1; Chemical Industry, Part 2.

Thursday, March 20

Morning: Symposia—Elevated Temperature, Utilities, Oil and Gas, Part 2; Chemical Industry, Part 3.

Afternoon: Symposia—Plastics, General Corrosion, Part 2; High Purity Water.

Friday, March 21

Morning: Symposia—Marine, Pulp and Paper, Aircraft Industry. Pipeline and Underground and General Corrosion Round Tables.

Four Round Tables Set for Conference

In addition to 16 symposia, four round table sessions are scheduled during the 14th Annual Conference technical program. Formerly two round tables were held on pipe line and underground corrosion and on general corrosion problems. The third and fourth, added this year, are in conjunction with T-7 Corrosion Coordinating Committee and with T-8 on Refining Industry Corrosion committee.

They will be held as follows:

Wednesday morning, March 19, Civic Auditorium. Refining Industry Round Table. R. W. Manuel, Socony Mobil Oil Co., Moderator.

Wednesday morning, March 19, Civic Auditorium. T-7 and Underground Corrosion Coordinating Round Table. C. A. Erickson, Jr. The People's Natural Gas Co., Pittsburgh, chairman; C. L. Mercer, Southwestern Bell Telephone Co., Houston, co-chairman.

Friday morning, March 21, Civic Auditorium. General Corrosion Problems Round Table, A. S. Joy, Brumley-Donaldson Co., Huntington Park, Cal., chairman; Joseph Bigos, U.S. Steel Corp., co-chairman.

Friday morning, March 21, Civic Auditorium. Pipe Line and Underground Corrosion Round Table. J. N. Hunter, Jr., Service Pipe Line Co., Tulsa, chairman.

Turn to Page 75 for a Chronological List of Technical Committee Meetings at San Francisco

Only 20 Booths at San Francisco Show Are Not Allocated; New Exhibitors Named

All but 20 of the 120 booths laid out for the NACE 14th Annual Exhibition at San Francisco's Civic Auditorium have been taken. The exhibition will be open March 18-20 concurrently with the 14th Annual Conference.

Since publication of the supplementary list of exhibitors' names on Page 108 of CORROSION's December issue, ten more firms have reserved space. Other lists of exhibitors have been published in October, Page 112 and November, Page 126.

Added firms are listed below:

Armour Chemical Div., Armour & Company, Chicago, Ill.

Corning Glass Works, Corning, New York

Electro Rust-Proofing Corporation, Belleville 9, N. J.

Fisher Research Laboratory, Inc., Palo Alto, Cal.

Inertol Company, Inc., San Francisco, Cal.

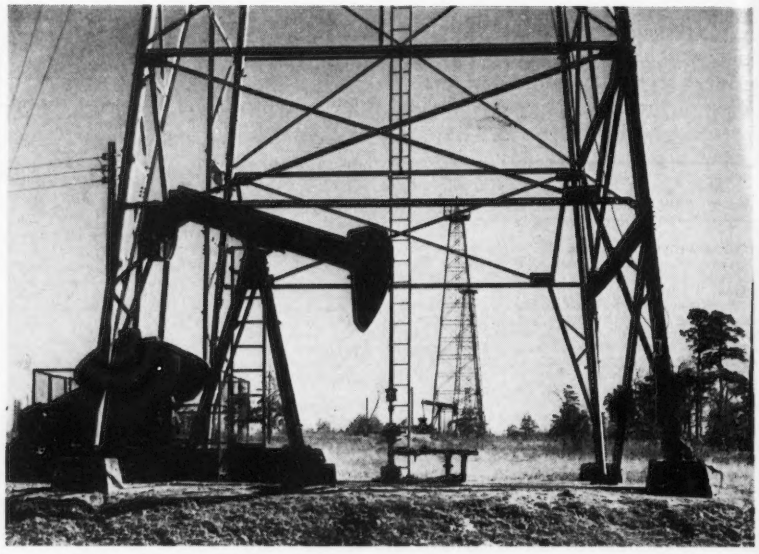
Polyken Sales Div., The Kendall Company, Chicago, Ill.

Phelps Dodge Copper Products Corporation, Los Angeles, Cal.

Royston Laboratories, Inc., Blawnox, Pittsburgh, Pa.

Shell Chemical Corporation, New York, N.Y.

Tinker & Rasor, San Gabriel, Cal.



IS CASING CORROSION A PROBLEM IN YOUR WELLS?

This case history tells how three companies stopped external casing corrosion on 70 of their oil and gas wells in Western Kansas. It was thought that Dakota water was the cause of most of the corrosion.

The companies called on CSI to survey the wells and determine the current requirements for cathodic protection. These ranged from 0.8 to 3.10 amperes. Then CSI was engaged to complete the job.

Galvomags, Dow's high-potential magnesium anodes, were installed to protect the casing. CSI furnished all engineering, labor, materials and installation equipment, including power augers and ditchers.

The cost averaged $350 per well—for installations designed to give 10 years of protection. The work took 30 days.

Call or write today for engineering or installation help with your corrosion problems—well casing, pipe line, etc. CSI also offers a complete line of quality cathodic protection materials at competitive prices. Estimates or quotations without obligation.

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Papers on Reinforced Plastics Will Be Given

Numerous Items on Agenda Related to Corrosion Problems

Numerous papers and discussions of interest to corrosion workers concerned with the application of reinforced plastic materials will be given during the February 4-6 Thirteenth Conference of the Society of the Plastics Industry, Inc., Reinforced Plastics Division. The meeting will be held at the Edgewater Beach Hotel, Chicago.

Among the items of interest scheduled are:

Thursday, February 4

Optimum High-Strength Epoxy Laminates, Harry Raech, Jr., Northrop Aircraft, Inc. and Fred Harris, Grand Central Rocket Co.

Effect of Curing Conditions on Physical Properties, Grant Brown, Cordo Chemical Corp.

Determination of Cure State in Reinforced Plastics by Dielectric Measurements, John J. Knudsen, Delsen Corp.

Improvement of Reinforced Plastics by Fibers from New Glasses, James W. Case, Dept. of Navy; Albert Lasday and J. Frees Brassy, Houze Glass Corp.

Mechanical Properties of Glass Reinforced Casting Resins, C. L. Segal, Hughes Aircraft Co.

Glass Fabric-Base Laminate Physical Properties as a Function of Finishing Variables, Richard C. Horton and Henry S. Falls, J. P. Stevens & Co., Inc.

Bond of Resin to Glass, A. C. Anderson and J. H. Healy, A. O. Smith Corp.

Long-Term Rupture Strength in Glass Reinforced Plastics, S. Goldfein, U. S. Army Engineer Res. & Dev. Lab., Fort Belvoir, Va.

Unidirectional Glass Reinforced Plastic Ring Structures, N. Chessin, General Electric Co.

Creep of Laminated Epoxies, John Delmonte, Furane Plastics, Inc.

Laminates Reinforced With Asbestos, Norman E. Wahl and Harold M. Preston, Cornell Aeronautical Laboratory, Inc.

Some Factors Influencing Durability of Glass Reinforced Laminates, Arthur L. Smith and John R. Lowry, Rohm & Haas Co.

Application of Reinforced Plastics to Transportation Uses, Alan Cripe, Chesapeake & Ohio Railway.

Reinforced Plastics in Architectural Applications, Attilio Forte and Warren Richardson, Holabird & Root & Burgee.

Effects of High Intensity Thermal Radiation on Plastic Laminates, H. S. Schwartz and B. J. Lisle, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

Effects of Elevated Temperature and Erosion on Reinforced Plastic Laminates, Norman B. Miller and Eric L. Straus, The Martin Co.

Behavior of Reinforced Plastics at Very High Temperatures, L. H. Shenker and I. J. Gruntfest, General Electric Co.

Wednesday, February 5

Fundamental Studies of Adhesion, J. J. Bickerman, Massachusetts Institute of Technology.

Resin-Glass Bond Characteristics, F. J. McGarry, Massachusetts Institute of Technology.

Behavior of Fibrous Glass Laminates From Interior Strain Measurements, Richard E. Chambers, Massachusetts Institute of Technology.

Effect of Polyester Resin and Process Variables on Laminate Properties, William G. Carson and Arthur L. Smith, Rohm & Haas Co.

Contour Weaving of Reinforcement, A. R. Campman and Edward Koppelman, Raymond De Icer & Engineering Co.

Thursday, February 6

Reinforced Plastic Transportation Equipment: A Bright Future, Kenneth C. Sanders, The Heil Co.

Development of Improved Filament Wound Pressure Vessels, George Epstein, Harry A. King and Robert H. Bandarzewski, Aerojet-General Corp.

Plastic Boats in the United States Navy, Cdr. C. L. Fears and Henry Shek, Dept. of Navy, Bureau of Ships.

Reinforced Plastics and Protective Coatings for Wood, Richard Mark, Balsa Ecuador Lumber Corp. and Bert M. Zuckerman, wood pathologist.

Anti-Corrosion Compound Is Developed in Hungary

Development of a successful anti-corrosive compound called "ferrofixol" is claimed by the Research Institute for Heavy Chemical Industry (Nehezvegyipari Kutato Intezet) at Veszprem, Hungary, some 60 miles southwest of Budapest. The research laboratories, established in 1950-52, concentrate largely on research associated with fertilizers.

A new method was developed for treatment of rock phosphates which produces the fertilizer "Nifos" which has high resistance to hygroscopic caking.

The institute has produced also refractory bricks from indigenous materials and a structural material from blast furnace slag, a substitute for acid and wear-resistant steel. Considerable work is done also in uses for coal, especially in production of gas, and exploitation of the coal tar chemical residues from coking operations.

The institute functions as a site for examination of the theoretical and economical aspects of industrial chemical operations, operates pilot plants and supplies technicians as consultants in the heavy chemical and stray of Hungary.

Details of the institute's operations were supplied in an English-language supplement to a well-printed and illustrated booklet in German.

BOOK NEWS

Prace Konferencji Elektrochemicznej. (In Polish, Russian and German) 890 pages, 6½ x 9¼ inches, cloth. 1957. The Institute of Physical Chemistry of the Polish Academy of Sciences, Warsaw, Poland. Availability not indicated.

Reports delivered at the November 1955 electrochemical conference held in Warsaw under auspices of the Polish Academy of Sciences.

Atomic Energy Facts. 216 pages, 8 x 10¼ inches, paper. September, 1957. United States Atomic Energy Commission. Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Per copy, $2.

A summary of atomic activities of interest to the industry. The book is identified as the first in a series of books sponsored by the AEC as a part of its industrial information program.

Contents include a discussion of the pattern of atomic energy, economic and manpower requirements, effect on industry, the work of government in the atomic energy program, licenses, patents and industry and international atomic cooperation.

Organization and functions of the AEC are described. Production of uranium and thorium is described with some information on locations of principal ore deposits. Power reactors are extensively considered with data on costs, design, experimental program, fuel cycles, fuel systems, separation processes, waste disposal, use of fission products.

Reactor materials are considered for heavy water, with special attention to reactor grade graphite production, beryllium and zirconium. Characteristics of research reactors are detailed and the development and use of isotopes considered.

Appendices give categories of restricted data available to holders of access permits, depositories of atomic data, rules of the commission and extensive tabulated data on power reactors.

Tin "Whiskers" Grown for Superconductivity Tests

Tin "whiskers" were grown rapidly from the polished edges of a stack of tin-plated steel squares when a pressure of several thousand pounds per square inch was applied to the top of the stack. This technique produced filaments about 0.02-inch long and about 0.0001-inch in diameter for studies on superconductivity at the National Bureau of Standards. The experiments showed that a magnetic field restores resistance to the "whisker" while in a superconducting state.

British Scientist Sees Corrosion as Definite Branch of Science

"Corrosion is coming increasingly to be recognized as a definite branch of science—namely, that which deals with the reactions between metals and their non-metallic environments." This quotation from the lecture "metallic Corrosion and Conservation," by W. H. J. Vernon, consultant, before the Institution of Civil Engineers, 1957, indicates that Mr. Vernon believes corrosion to be a study rewarding specialization.

Mr. Vernon also comments extensively on the various corrosion processes involved in electrolytic and metal-gas reactions as well as the various kinds of indigenous mechanisms limiting corrosion on some materials. Consideration is given also to enumeration of various principles that are involved: viz, principle of the controling factor, of critical humidity, and others.

Principles involved in the various methods adopted to mitigate corrosion are defined and considerable attention is paid to the economics of corrosion prevention, including painting and cathodic protection. Aggregate costs of corrosion are also enumerated, including an estimate of corrosion losses in the United Kingdom for 1955 of 406 million pounds sterling.

Numerous data are given also on the value of alloys used for corrosion prevention, cost of equipment deterioration, including motor vehicles and some costs in specific industries and public services.

WORLD WIDE NEWS IN CORROSION

In the General News Section of CORROSION items about the corrosion control activities of organizations all over the world are published. This permits CORROSION readers to keep abreast of events abroad as well as those in the United States. During recent months items have been published about Belgium, France, England, Italy, Japan, The Netherlands, Germany, Poland, Roumania, Russia and other countries.

TECHNICAL REPORTS

on

Corrosion in Oil and Gas Well Equipment

T-1A Survey of Corrosion Control in California Pumping Wells. A Report of T-1A on Corrosion in Oil and Gas Well Equipment, Los Angeles Area. Pub. 54-7. Per Copy, $.50.

T-1C Current Status of Corrosion Mitigation Knowledge on Sweet Oil Wells. A Report of Technical Unit Committee T-1C on Sweet Oil Well Corrosion. Per Copy, $.50.

T-1C Field Practices for Controling Water Dependent Sweet Oil Well Corrosion. A Report of Technical Unit Committee T-1C on Sweet Oil Well Corrosion, Compiled by Task Group T-1C-1 on Field Practices. Pub. No. 56-3, Per Copy $1.00.

TP-1D Sour Oil Well Corrosion. *Corrosion* August, 1952, issue. NACE members, $.50; Non-members $1 Per Copy.

TP-1G Field Experience With Cracking of High Strength Steel in Sour Gas and Oil Wells. (Included in Symposium on Sulfide Stress Corrosion.) (Pub. 52-3) $1 Per Copy, 5 or more copies to one address, $.50 Per Copy.

T-1G Sulfide Corrosion Cracking of Oil Production Equipment. A Report of Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking. Pub. 54-5. $.50 Per Copy.

T-1G Hydrogen Absorption, Embrittlement and Fracture of Steel. A Report on Sponsored Research on Hydrogen Sulfide Stress Corrosion Cracking Carried on at Yale University, Supervised by NACE Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking by W. D. Robertson and Arnold E. Schuetz. Pub. 57-17. Per copy $2.

T-1J Status Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Per Copy $1.00.

T-1J Reports to Technical Unit Committee T-1J on Oilfield Structural Plastics. Part 1, Long-Term Creep of Pipe Extruded from Tenite Butyrate Plastics. Part 2, Structural Behavior of Unplasticized Geon Polyvinyl Chloride. Publication 55-7. Per Copy $.50.

T-1J Summary of Data on Use of Structural Plastic Products in Oil Production. A Status Report of NACE Technical Unit Committee T-1J on Oilfield Structural Plastics. Per Copy $.50.

T-1J Service Reports Given on Oil Field Plastic Pipe. Activities Report of T-1J on Oilfield Structural Plastics. Per Copy $.50.

T-1J Oilfield Structural Plastics Test Data are given. Activities Report of T-1J on Oilfield Structural Plastics. Per Copy $.50.

T-1J Reports to Technical Unit Committee T-1J on Oil Field Structural Plastics. Part 1—The Long Term Strengths of Reinforced Plastics Determined by Creep Strengths. Part 2—Microscopic Examination as a Test Method for Reinforced Plastic Pipe. Per Copy $.50.

T-1J Status Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Part 1—Laboratory Testing of Glass-Plastic Laminates. Part 2—Service Experience of Glass Reinforced Plastic Tanks. Part 3—High Pressure Field Tests of Glass-Reinforced Plastic Pipe. Per Copy $.50.

T-1J Service Experience of Glass Reinforced Plastic Tanks—Status Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Pub. 57-18. Per Copy $.50.

T-1J Experience with Oil Field Extruded Plastic Pipe in 1955—A Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Pub. 57-19. Per Copy $.50.

T-1K Proposed Standardized Laboratory Procedure for Screening Corrosion Inhibitors for Oil and Gas Wells. A Report of T-1K on Inhibitors for Oil and Gas Wells. Publication 55-2. Per Copy $.50.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

1061 M & M Bldg. Houston 2, Texas

British Corrosion Group Meetings Are Scheduled

Meetings of the Corrosion Group, Society of Chemical Industry scheduled for the first 5 months of 1958 are:

Jan. 15—Stress corrosion Cracking, T. P. Hoar and J. G. Hines, Society of Chemical Industry, 14 Belgrave Square, London, S. W. I. 6:30 pm.

Jan. 23—Discussion. Exhibition of protective coatings. Battersea College of Technology, Battersea Park Road, London, S. W. 11. 6:30 pm.

Feb. 17—Protection of Spraying and Dusting Equipment, Representatives of the National Institute of Agricultural Engineering, (Joint meeting with pesticides group). Society of Chemical Industry. 6 pm.

Feb. 26—Stress Corrosion Cracking of Stainless Steels, T. P. Hoar and J. G. Hines. Stress Corrosion Cracking of Carbon and Low Alloy Steels, R. N. Parkins. (Joint meeting with Newcastle Section). Stephenson Bldg., King's College, Newcastle-upon-Tyne. 2:30 and 6 pm.

March 11-12—Symposium on Protection of Motor Vehicles. Corrosion of Cylinders and Moving Parts of Engine, W. G. Stevenson. Corrosion Inhibitors for Coolant, A. T. B. P. Squires. Research and Experience With Sodium Benzoate and Sodium Nitrite Mixtures as Corrosion Inhibitors in Engine Coolants, A. D. Mercer and F. Wormwell.

Oil Additives, N. E. F. Hitchcock. Protection of Accessories, A. A. Tomkins. Packaging of Vehicles and Components for Export, R. J. Brown. Experience With the Land Rover, S. Heslop. Phosphate Treatment of Vehicle Bodies, G. Murray. Experience With Aluminum Alloys in Motor Vehicles, F. C. Porter. 9:30 am and 2:30 pm.

March 28—Recent Research on the Corrosion of Boiler Tubes, E. C. Porter (Joint meeting with Manchester Section) Robinson Lecture Theater, The University, Manchester. 6:30 pm.

April 16—Annual general meeting and spring lecture. Corrosion Research and Its Industrial Background, W. H. J. Vernon. Society of Chemical Industry, London. 6:30 pm.

April 17—Annual general meeting of educational panel. Society of Chemical Industry, London. 11 am.

Members of the National Association of Corrosion Engineers are invited to be present at these meetings.

pH Standards Defined From 0 to 95 C by NBS

V. E. Bower and R. G. Bates of the National Bureau of Standards' physical chemistry laboratories have completed definition of the standard for pH in temperatures from 0 to 95 C. This extends the standard beyond the 60 C limit formerly established. Because computation of pH is based on several reasonable assumed relationships between ionic and mean activities, the electromotive force of cells employing the standards as electrolytes was measured using highly reproducible hydrogen and silver-silver chloride electrodes.

The work was based on a recent determination of the standard potential of the silver-silver chloride electrode, which included measurements from 0 to 95 C. A report on the work was published in *J. Res. Nat. Bur. Standards*, **59**, 261 (1957) Oct. RP 2797.

Using impressed current anodes?

NATIONAL
TRADE-MARK

GRAPHITE ANODES ARE BEST!

FOR LOW COST...

"National" graphite anodes provide ample conductivity for economical ground bed design in both low and high resistance soils. Installed with graphite particle or coke backfill, "National" anodes offer low initial cost; exceptionally long life.

National Carbon manufactures a complete line of anodes providing the economies of graphite to all types of cathodic installations.

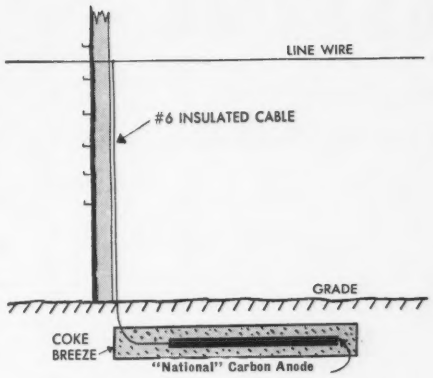
3″ x 60″ and 4″ x 80″
sizes in plain and NA Graphite

3″ x 30″ and 3″ x 60″
NA Graphite Anodes with Type QA Connections

2″ x 12″, 2″ x 20″ and 3″ x 30″
Type QA Anodes of NA Graphite

If you do not use "National" Graphite Anodes for cathodic protection, try them! We believe you'll find "NATIONAL" GRAPHITE ANODES ARE BEST FOR LONG LIFE AND LOW COST!

FOR LONG LIFE...



Since 1943, a distributed "National" carbon anode system has protected 2042 feet of 4 inch bare steel main for a large Eastern utility company. Installed horizontally (see diagram) to avoid shale rock, 16 — 2″ x 80″ anodes are spaced approximately 120 feet apart to obtain uniform protection.

Before installation of the "National" carbon anodes, 4 leaks occurred on the main in one year. Since cathodic protection began in October, 1943, there has not been a single leak.

• • •

INSTALLATION DATA

Anode size: "National" 2″ x 80″ Carbon
Backfill: Coke breeze
Length of anode service: 15-30 ampere-years per anode
Average soil resistance: 1000 to 10,000 Ohm. cm.

The terms "National" and "Union Carbide" are registered trade-marks of Union Carbide Corporation

UNION CARBIDE

NATIONAL CARBON COMPANY • **Division of Union Carbide Corporation** • ***30 East 42nd Street, New York 17, N.Y.***
SALES OFFICES: Atlanta, Chicago, Dallas, Kansas City, Los Angeles, New York, Pittsburgh, San Francisco • **IN CANADA:** Union Carbide Canada Limited, Toronto

A NEW DEGREE OF RESISTANCE TO CHEMICAL CORROSION

Tropical Paint Company's

Tropoxies

Protect Wood, Metal and Masonry Surfaces

On "problem" surfaces, where chemical fumes or liquids quickly break down ordinary paints, new Tropoxy enamels provide superior, longer-lasting protection. These new epoxy-resin coatings effectively retard chemical corrosion on exterior metal, wood, concrete, plaster, brick, cement or cinder block. Multiple coats can be applied within hours to achieve ideal 5-mil thickness.

PROTECTIVE COATINGS for CORROSION PROBLEMS

Also investigate our oleoresinous phenolics, rubber-base paints and enamels, high-heat (up to 1000°) aluminums. They have been performance-proved on thousands of applications.

FREE — complete information on our specialized industrial coatings sent on request.

TROPICAL PAINT CO.

1142-1204 W. 70th, Cleveland 2, Ohio
Heavy-Duty Maintenance Paints Since 1883

Military Problems Are Posed for Scientists

Military scientists are seeking the following: A gamma radiation shielding material with greater mass absorption coefficient than but lighter than lead; light metals and alloys with Young modulus value of 30 million psi and close to structural steel in properties but much lighter for breech rings, bolts, barrels and other ordnance.

Low alloy steel suitable for gun tubes; lightweight material with resistance to abrasion and erosion of high strength steels; magnesium alloys as corrosion resistant as steel and preferably of comparable cost; stainless metal with properties and approximate cost of low-alloy steel; lightweight, high strength material for gun barrels; high temperature (10,000 C or over) magnetic materials.

Scientists interested may get copies of "Inventions Wanted" from National Inventors Council, U. S. Department of Commerce, Washington, D. C.

200 Papers on Nuclear Reactors Set at Chicago

More than 200 papers on various aspects of atomic science will be presented during the March 17-21, 1958, Nuclear Congress to be held at International Amphitheater, Chicago. Over 30 engineering and scientific societies are participating in the program, according to John W. Landis of The American Nuclear Society, Inc., chairman of the program committee.

Among others, sessions have been scheduled on reactors for process heat and radiation, reactor operation and maintenance, experimental power reactors and advanced concepts, thermal and mechanical design, reactor plant materials, water contamination and treatment and shielding and containment.

Stress-Free Multi-Phase Metals Are Impossible, NBS Tests Indicate

Impossibility of obtaining completely stress-free condition in metals which contain more than one phase were shown in X-ray diffraction experiments conducted at National Bureau of Standards by C. J. Newton and H. C. Vacher. The experiments indicate that textural stresses resulting from different thermal expansion coefficients of phases must be taken into account in any study of the metal's reaction to its environment, as in corrosion studies, fatigue or crack formation.

Details of the work are reported in "An X-ray Study of Textural Stresses in Two-Phase Alloys," *J. Res. National Bureau of Standards,* **59,** No. 4 (1957) RP 2793.

Architectural Metal Meet

The 20th Annual Convention of the National Association of Architectural Metal Manufacturers will be held April 27-May 2 at the Shamrock Hilton Hotel, Houston.

Air and Water Pollution Prevention Sessions Set

Preventing Air and Water Pollution is the title of a session to be held at 9:15 a.m. Wednesday, January 29 during the Plant Maintenance and Engineering Show and Conference at Chicago. It is one of 25 sessions to be held during the event scheduled to be held January 27-30 at the Chicago Amphitheater and Palmer House.

Speakers will be Harry Ballman, Bituminous Coal Institute, Washington, D. C., formerly executive secretary, Air Pollution Control Association, Pittsburgh, who will discuss air pollution, and Edward J. Cleary, executive director and chief engineer, Ohio River Valley Water Sanitation Commission, Cincinnati, who will discuss water polution.

Liege Meeting to Cover Corrosion by Water

A session will be held June 10-11 at Liege, Belgium, by Centre Belge D'Etude et de Documentation des Eau to consider control of corrosion by water and in aqueous media. Topics to be considered are fundamentals, rust and sediments, corrosion by industrial water, materials, soils and bacteria, marine environments and corrosion in boilers and in gas streams.

The session will be held during the Cebedeau world congress for the study of water. Other sessions will be devoted to industrial use of water and purification of waste waters. The final session on June 14 will concern the future outlook on the use of water by industry and control of water-connected corrosion.

Gas Turbine Technical Sessions Are Scheduled

Technical information on design, operation and future engineering prospects of gas turbines, free-piston engines and their components and accessories, including Diesel superchargers will be discussed March 3-6 at the Shoreham Hotel, Washington, D. C. by the Gas Turbine Power Division of the American Society of Mechanical Engineers.

A wide variety of data on actual operating experience and theoretical considerations related to the gas turbine will be presented in technical sessions.

$30 Millions Allocated for Fundamental Research

More than $15 millions is being put into the 30-year-old fundamental research program of E. I. duPont de Nemours & Co., Inc., in the search for scientific knowledge without specific commercial objectives. The company's research encompasses inquiries into organic chemistry, biological chemicals, polymers and plastics, fibers and films, metals and alloys, high purity elements for electronic uses and for solar energy conversion, radiation chemistry and others.

•

South Central Region NACE meets in New Orleans in 1958.

•

The 1957 alphabetical author index to CORROSION's Technical Section includes 244 names.

Carnegie Laboratory Marks 25th Anniversary

The 25th anniversary of the founding of the Metals Research Laboratory of Carnegie Institute of Technology was held October 24.

Among the four prominent metallurgists appearing on the technical program held during the celebration was Julius J. Harwood, Head, Metallurgy Branch, Office of Naval Research.

Radiation Effects Center

A Radiation Effects Information Center has been established by the U. S. Air Force at Battelle Memorial Institute, Columbus, to gather and disseminate data concerning effects of nuclear radiation on materials and systems required in aircraft.

TECHNICAL REPORTS

on

PIPE LINE CORROSION

T-2 Statement on Minimum Requirements for Protection of Buried Pipe Lines. Prepared by a Special Task Group of NACE Technical Group Committee T-2 on Pipe Line Corrosion. Publication No. 56-15. Per Copy $.50.

TP-2 First Interim Report on Galvanic Anode Tests. (Pub. 50-2) NACE members, $3; Non-members, $5 Per Copy.

T-2B Final Report on Four Annual Anode Inspections. A Report of Technical Unit Committee T-2B on Anodes for Impressed Current. Publication 56-1. Per Copy $1.00.

T-2B Use of High Silicon Cast Iron for Anodes. First Interim Report of Unit Committee T-2B on Anodes for Impressed Current. Publication No. 57-4. Per Copy $.50.

T-2C Some Observations of Cathodic Protection Potential Criteria in Localized Pitting. A Report of T-2C on Minimum Current Requirements for Cathodic Protection. Pub. 54-2. Per Copy $.50.

T-2C Some Observations on Cathodic Protection criteria. A contribution to the work of NACE Technical Unit committee T-2C. Publication No. 57-15. Per Copy $.50.

T-2G Tentative Recommended Specifications and Practices for Coal Tar Coatings for Underground Use. A Report of Technical Unit Committee T-2G on Coal Tar Coatings for Underground Use. Per Copy $.50.

T-2H First Interim Report. Tentative Recommended Specifications for Asphalt Type Protective Coatings for Underground Pipe Lines—Wrapped Systems. A Report of NACE Technical Unit Committee T-2H on Asphalt-type Pipe Coatings. Publication No. 57-11. Per Copy $.50.

T-2H Second Interim Report. Tentative Recommended Specifications for Asphalt-type Protective Coatings for Underground Pipe Lines—Mastic Systems. A Report of NACE Technical Unit Committee T-2H on Asphalt-type Pipe Coatings. Publication No. 57-14. Per Copy $.50.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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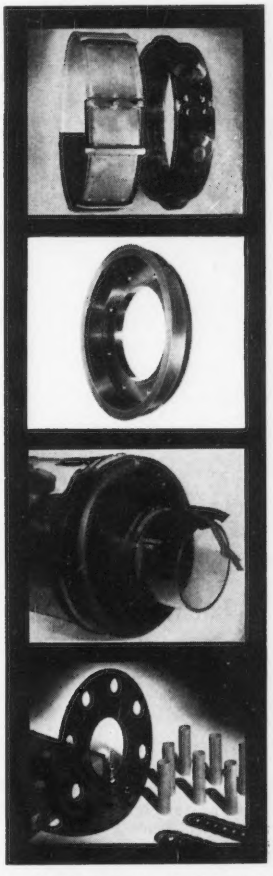

CROSSING INSULATORS

Truly rugged crossing insulators, the Maloney Models 55 and 57 shown are in addition to the wrap-around Model 56 crossing insulator in the complete line developed in the Maloney Research and Development Laboratory. In crossing insulators, as in other products, Maloney research has set *new* industry standards.

SCRAPER CUPS

The unique lip design of the Maloney scraper cup insures complete effectiveness throughout the life of the cup. Completely adaptable to out-of-round and other adverse conditions, the Maloney scraper cup *has no equal*. If time is a factor, F. H. Maloney is *'within arm's length.'*

CASING SEALS

This zipper type casing seal is equally suited for new or replacement applications. The zipper joint assures a dust-tight, water-tight seal — installs in minutes. Slip-on types for new applications are also available. *Both* compensate for eccentric installations without any special handling, and are available to you *immediately*.

FLANGE INSULATION

Maloney Flange Insulation sets include all components for each joint; central gasket, sleeves, and washers of linen base Bakelite. High pressure applications are available with a Neoprene faced central gasket — *eliminating* any other gasket seal. Another result of *continuous* Maloney research.

SETTING *NEW* STANDARDS FOR THE INDUSTRY

F. H. Maloney COMPANY

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Equipment Services NEW PRODUCTS Materials Literature

Adhesives

Ray-Bond R-84001, a hot melt adhesive which has instant grab when applied to etched Teflon, Mylar, polystyrene, polyethylene and almost any other material has been developed by Raybestos-Manhattan, Inc., Bridgeport 2, Conn. While intended for temporary fastening in assembly operations the material will make a permanent bond if temperatures do not exceed 150 F.

Atomic Energy

Radiation Cells can be built underground at low cost through techniques developed by Applied Radiation Corporation, 2404 N. Main St., Walnut Creek, Cal. A chamber for a 30 kw 50 million Ev linear accelerator was constructed at a cost of $54,000 using high density, lead ore slag as main shielding. A 40-ton concrete door blocks entrance to the cave chamber.

•

Silicone-Insulated electric motors recently subjected to gamma ray radiation at a rate of 0.1 megarads an hour over extended periods while operating at 200 C are expected to last 10 years under test conditions, according to Dow Corning Corp., Midland, Mich. Test data confirms the tendency of radiation to atack side-groups of a long chain molecule or polymer, promoting cross-linkage and stiffening. Because cured silicones are almost completely cross-linked already, radiation has little effect.

Batteries

Gulton VO-Series nickel cadmium batteries, among others will be produced by a new Alkaline Battery Division of Gulton Industries, 212 Durham Ave., Metuchen, N.J. The company has been licensed to produce and market in the United States batteries made under patents held by SAFT-France.

•

Silent Power is made in a new fuel cell using hydrogen and oxygen as power developed by National Carbon Company, Division of Union Carbide Corp., 100 East 42nd St., New York 17, N.Y. First significant application of the principle is in the new "Silent Sentry" using this power source, made for the U.S. Army Signal Corps. The small, portable radar set is designed to give intelligence on combat operations in spite of smoke, darkness or fog. The fuel cell, using hollow carbon electrodes, produces electrical energy through the reaction between hydrogen and oxygen and has an efficiency of about 70 percent, twice that of conventional thermal systems producing electrical current. One killowatt of power can be produced from a cell with a volume of one cubic foot. Voltage across the electrodes is about one and desired voltage can be obtained by connecting batteries in series. Cells now used operate at from approximately atmospheric pressure 120 to 140 F. At higher pressures increased output can be obtained. Amount of current is in proportion to physical size of the cell, It can be operated using hydrogen and air, including industrial grade of hydrogen including impurities.

Consideration is being given to harnessing the new batteries to utilization of solar energy, in which the sun's heat is used to separate the components of water and also by using the hydrogen and oxygen developed in nuclear power plants. Presently efforts are made to suppress hydrogen and oxygen evolution in nuclear plants. An economical source of hydrogen and oxygen would make operation of the batteries economically practical in many instances.

Chemicals

Physical Properties of Synthetic Organic Materials, a 28 page booklet (F-6136) is available from Union Carbide Chemicals Co., 30 East 42nd St., New York 17, N. Y. The booklet lists latest physical property data on more than 350 organic chemicals, including 50 new chemicals introduced by the company since the previous edition of the book was published. An alphabetical index is included.

Coatings

Anodized finishes on metals produced by the Hardas Process are described in a brochure available from Anachrome Corp., 10647 Garfield St., South Gate, Cal. The firm specializes in hard anodizing and precision plating.

•

Endura-Temp, a suspension coating containing ceramic frit is designed for use on iron and steel at temperatures up to 1500 F. The air-drying coating may be sprayed, brushed or dipped on a clean surface without primer. More information can be obtained from Products Development Corp., 824 Connecticut Ave., N.W., Washington 6, D.C.

•

Iso-Lok ZDP, a conversion coating for zinc plating which produces a bright finish without leaching is designed for service where resistance to corrosion is more important than appearance. More data are available from Wagner Brothers, Inc., 400 Midland, Detroit, Mich.

•

Mono-Seal, a coating consisting of a blend of silicones and epoxies, contains no plasticizers or oils, cures by combined solvent evaporation and polymerization and produces a coating which is elastic, well-bonded and durable, according to Mono-Seal Products, 44 Garden St., Everett 49, Mass.

Heating Elements

Partlow Corp., 505 Campion Rd., New Hartford, N.Y. has published Bulletin 110, which lists over 400 corrosive atmospheres into which temperature control elements often are immersed, together with recommended bulb materials for these environments.

Truflex corrosion resistant thermostat metals are described in TRU-6, a 4-page technical bulletin titled "Corrosion Resistant Thermostat Metals," available from General Plate Division, Metals and Controls Corp., Attleboro, Mass.

Inhibitors

PQ No. 500, an inhibitor emulsifiable with water or suitable for mixing with solvents or oils is recommended by American Oil and Supply Co., 238-60 Wilson Ave., Newark, N.J. for long and short-term protection of metals not exposed to outside atmospheres. The material, of unspecified composition, may be mixed in varying proportions with water to form an inhibitive liquid whose effectiveness is directly proportional to the concentration of inhibitor in the mixture, as rated by salt spray and humidity cabinet tests. The mechanism described indicates the inhibitor preferentially wets metal surfaces permitting water to evaporate.

•

Stimulation of the flow of oil wells by application of inhibited hydrochloric acid was first tried in a Michigan oil well in the winter of 1932 by technicians of Dow Chemical Co. and Pure Oil Co. Dowell, Inc. was formed the following November to exploit the method, and it has been active in well acidizing and other well services during the succeeding 25 years. More than 375,000 oil and gas wells have been treated by the company, to produce an additional estimated 2 billion barrels of oil that otherwise would have been lost.

•

Emocithin, a water-in-oil emulsifier, developed by W. A. Scholten's Chemische Fabrieken, Foxhol, Netherlands and distributed in the United States by Morningstar, Nichol, Inc., 630 West 51st St., New York, N.Y., produces emulsions which will not separate at temperatures up to 200 F. It is tasteless and odorless and may be used in food, pharmaceuticals or as a dispersing agent in dyes and paints. Data on its use in Europe is available.

Instruments

Transistorized computer-data systems have been developed by Beckman Instruments, Inc. Anaheim, Cal. for use in directing the operations of Phillips Chemical Company's Sweeny, Texas ethylene plant. The unit is said to be the first industrial data system with solid state components in place of vacuum tubes.

•

Instantaneous Analysis of sequential chemical reactions such as those which occur in explosion of rocket fuels can be made with a device produced by Cincinnati Division, Bendix Aviation Corp. The device, called a "Time-of-Flight Mass Spectrometer" is expected to have numerous applications in metallurgical and chemical processing operations. It can make a chemical analysis

(Continued on Page 115)

NEW PRODUCTS

(Continued From Page 114)

in 1/10,000 second, instantly identifying vaporized gases, liquids and solids (including metals) by revealing their respective molecular masses.

Laboratories

Water Service Laboratories, Inc. has leased 11,000 square feet of 169 West Wyoming Ave., Philadelphia for its branch office there.

•

Atlas Powder Company has established a Toxicology Section in its Chemical Research Department to investigate the safety of Atlas products.

Metals—Ferrous

Inconel and Inconel-X metal bellows are being produced by Fulton Sylphon Division, Robertshaw-Fulton Controls, Knoxville, Tenn. in sizes from 15/32-inch to 12 inches outside diameter and in single to four plies. They are useful in temperatures up to 1500 F.

•

Carbon and Alloy seamless steel tubing and electric resistance welded steel tubing produced in several grades by Ohio Seamless Tube Division, Copperweld Steel Co., Shelby, Ohio are described in a new general catalog, CS-58 available now.

Metals—Non-Ferrous

Manganese Ore deposits at Batesville, Ark., will be explored by American Potash & Chemical Corp. In 10,700 acres of land 100 miles northeast of Little Rock, the deposits, if economically feasible for development, will be exploited by a new company to be formed among owners.

•

New Hard Lead sheet's use for roofing and flashing is described in Lead Industries Association's brochure, Modern Sheet Lead for Roofing and Flashing.

•

Niobium of unusual purity and niobium alloys with high strength at high temperatures have been developed by Pigments Dept., E. I. duPont de Nemours & Co., Inc., Wilmington, Del. for use in jet engines, missiles and atomic reactors. A joint development agreement has been made with Thompson Products, a leading producer of jet engine parts and aircraft components. Niobium is noteworthy for its extreme ductility in the absence of nitrogen, hydrogen or oxygen. A one-pound ingot can be cold-rolled into foil 1/1000-inch thick without annealing. A principal use of the metal is in alloys used for turbine buckets operating at over 1650 F. It has a low neutron cross section. Alloys are among the most corrosion-resistant known.

Non-Metallics

Niagara Concrete Pipe, Ltd., St. Catharines, Ont. has been purchased by American-Marietta Co. It will build another plant at Toronto. The industrial boom accompanying the development of the St. Lawrence Seaway has

(Continued on Page 116)

THE POSITIVE WAY TO PREVENT CORROSION

proved for more than 4 years on Texas Gulf Coast

Here's a proven corrosion-resistant coating that withstands the onslaughts of acids, caustics or salt air for longer periods with just two coats. Permaspray can be rapidly applied by either spray, brush, or roller, and requires about 4 hours drying time for normal conditions. The second coat can be applied without changing scaffolding. One gallon of Permaspray will cover 400-500 sq. ft. depending on the surface.

As an example of Permaspray's corrosion-resistance, the fan covering on the gas blower handling 20% hydrochloric acid in the waste recovery unit of a Gulf Coast plant withstood weather and acid for 3 years before sign of breakdown began to appear. Here are the results of four metal test panels, 4" x 8", sand blasted and coated with one coat of primer and 2 coats of Permaspray:

#1 Submerged in bay at Galveston, Texas
After 220 days — coating excellent

#2 Submerged in concentrated hydrochloric acid
After 240 days — excellent

#3 Submerged in caustic sodaflakes
After 275 days — excellent

#4 Submerged in fuel oil
After 275 days — excellent

Write today for folder giving details, including test with various acids, and prices.

permaspray manufacturing corporation

P. O. BOX 875 • LEAGUE CITY, TEXAS

NEW PRODUCTS

(Continued From Page 115)

created an unusually heavy demand for large sized concrete drainage pipe.

•

Halocarbon 437 (2,2,3-Trichloroheptafluorobutane), a clear, colorless liquid at room temperature is highly resistant to oxidizing agents, non-flammable and non-polar. Its good heat transfer characteristics recommend it for use as a coolant or transfer medium, Hacocarbon Products Corp., 82 Burlews Court, 82 Burlews Court, Hackensack, N. J. claims.

•

Industrial Carbon products are produced in 8 minutes in a new two-step process in which the material is simultaneously subjected to an electrical current and to pressures of several tons per square inch. Methods formerly used called for mixing the materials, heating and extruding them and then baking shapes for several weeks in gas-fired furnaces.

•

Plastics

Teflon Bellows with twice the bursting strength and 20 times the flex life of conventional machined bellows are now being molded from a patented high density virgin Teflon compound by Resistoflex Corp., Roseland, N.J. The molding process maintains linear orientation throughout the bellows' convolutions and imparts optimum strength and fatigue life. Dynamic working pressures as high as 120 psi, full vacuum service and bending deflection up to 70 degrees and axial movements to 2 inches or more are characteristics.

•

Teflon insulated silver plated copper wire is being made by Halocarbon Division, Haveg Industries, Inc., Wilmington 8, Del. It is capable of continuous service up to 260 C and comes in 15 code colors in sizes 10 to 30.

•

Teflon 100-X perfluorocarbon resin shapes and filaments are being made in limited quantities by United States Gasket Co., Plastics Division of the Garlock Packing Co., Camden, N.J. The material has essentially the same characteristics as other Teflons except a top temperature service limit of about 400 F.

•

Kemlon Pumps, made of fluorocarbons, have a barrier diaphragm separating all working parts from the fluid handled. All surfaces of the pump in contact with corrosive are coated with Teflon, or may be made of corrosion resistant metals. They are manufactured by Keystone Engineering Co., P.O. Box 14366, Houston 21, Texas.

•

Titeflex, Inc., Springfield 4, Mass. has available a 4-page catalog supplement describing the new 110 Teflon hose assembly recently developed. The hose, with flexible Teflon innercore and stainless steel wire braid is equipped with new fittings suitable for systems operating up to 1500 psi and between—100 and 500 F.

•

Hetron 92, fire retardant, fiber glass reinforced polyester resin panels are offered by Resolite Corp., Zelionople, Pa. They are useful as substitutes for glass in locations where corrosive atmospheres or heat result in heavy breakage or damage.

•

Valves

Insertable Union check valves with Nylon bodies are described in Bulletin NP-25 available from Jordan Industrial Sales Div., OPW Corp., 6013 Wiehe Rd., Cincinnati 13, Ohio.

•

Glass Reinforced Polyester plastic valves manufactured by W. G. Rovang and Assoc., Inc., 1945 East Columbia Blvd., Portland 17, Oregon have high resistance to corrosives. Descriptive literature is available.

•

Welding

Arcos Corp., 1500 South 50th St., Philadelphia 43, Pa. describes in its brochure "EB Weld Insert" the use of consumable inserts to obtain a sound root welding pass when only one side of the weld is available. Fifteen compositions of inserts are available.

•

Welding Stainless pipe is simplified through the use of an aligning connector available from Speedline Div., Horace T. Potts Co., 504 Erie Ave., Philadelphia, Pa. It fits over ends of connecting sections permitting in-place assembly of pipe and fittings and helps to align the system.

MEN in the NEWS

Robert A. Woofter, formerly Coating and Corrosion Research Supervisor, Jones & Laughlin Steel Corp., has been promoted to metallurgical contact engineer. He has been with the firm since 1936.

•

Edward E. Finney has joined Pittsburgh Coke & Chemical Company as a sales representative in the firm's industrial chemical division.

•

Burton S. Payne, Jr., has been made metallurgical research and development group head in the research division.

•

Adrian R. Fisher has become chief executive officer of Johns-Manville Corp. **Clinton B. Burnett** was elected executive vice-president and made a director.

•

Arthur R. Simpson has been assigned to Chicago Bridge and Iron Company's Chicago sales office; **James R. Mansell** has been transferred to the Houston sales office from Birmingham and **George M. Rowley** has been moved from New Jersey to the Boston sales office.

•

E. E. Fogle has been made president of Union Carbide Chemicals Co.; **H. D. Kinsey** is president of the newly formed Union Carbide Olefins Co. which will handle production of ethylene, propylene, butadiene and other hydrocarbon products.

•

James W. Hakeman, formerly senior corrosion engineer for Tretolite Company at Shreveport, La., has been named assistant to the Rocky Mountain Division manager and will be located at Edmonton, Alberta. **James H. Burrell, Jr.,** has been named assistant to John Hampton, manager of West Central Division with headquarters in Midland, Texas. **Ellis Lee Ganey, Jr.,** is now field engineer at Jena, La, while **James P. O'Connor** is a corrosion engineer at the company's New Orleans office.

•

Gerald L. Moran has been appointed

(Continued on Page 117)

HEIL'S EXPERIENCE can help solve YOUR Equipment - Corrosion Problems

Laboratory facilities back Heil quality.

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MEN in the NEWS

(Continued From Page 116)

general manager of the Chemical and Metallurgical Division of Sylvania Electric Products, Inc.

•

Dr. Charles Henry Snow, dean emeritus of the College of Engineering, New York University, died October 28 at the age of 94.

•

Jeremiah J. Sullivan, Jr., has been named sales engineer for Taylor Fibre Co., Norristown, Pa., manufacturer of vulcanized fiber and laminated plastics.

•

Lloyd R. Anderson, chief metallurgist of Denver Division, Gardner-Denver Co., has been named assistant manager of the division. **Richard F. Schaffer** has been named chief metallurgist of the company.

•

K. H. Rowland has been appointed vice-president—Production of Union Carbide Chemicals Co. and **L. K. Merrill** has been made vice-president—technical of Bakelite Company, both divisions of Union Carbide Corp.

•

Carl E. Riestle, executive vice-president of Humble Oil & Refining Co., Houston, has been named to receive the Anthony F. Lucas Gold Medal for distinguished achievements in petroleum engineering by the American Institute of Mining, Metallurgical and Petroleum Engineers.

•

Morley H. Mathewson has been made director of industrial engineering of International Minerals and Chemical Corp., 20 N. Wacker Drive, Chicago 6.

•

Frank G. Weeden, Jr., has been appointed director of research and **Clarence Hester** has been made a sales engineer for John L. Doré Co., Houston.

•

John F. Thompson, chairman of the board of The International Nickel Company of Canada, Ltd. will receive the Charles F. Rand Memorial Medal for 1958 from American Institute of Mining, Metallurgical and Petroleum Engineers.

•

Frank Dorr has been made chief corrosion engineer for Standard Magnesium Company, Tulsa, with headquarters in the company's recently opened field office in the Bermac Bldg., 4101 San Jacinto, Houston.

•

C. P. Dillon, corrosion group leader at Union Carbide Chemicals Company plant at Texas City since 1950 will transfer in February to the company's Institute, W. Va. plant as materials engineer in the engineering department. He joined Union Carbide in 1941 as laboratory assistant at the company's South Charleston plant.

•

Ted Du Mond, formerly editor in chief of Materials and Methods magazine is new editor in chief of the American Society for Metals' Engineering Institute's educational courses. Specialized correspondence courses and in-plant instruction for metals engineer is offered by the new division.

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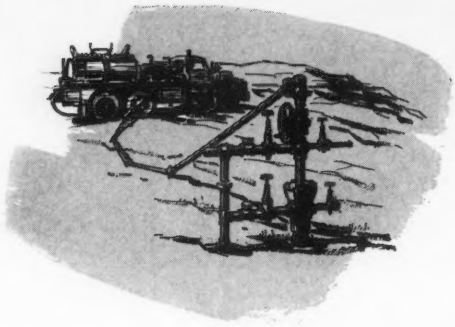

REPORT ON A NEW CONCEPT IN CORROSION CONTROL

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So far, results have been outstanding. The following case history is an example of the results obtained with this new method.

A gas-lift well in South Texas was producing 25 BOPD and 85 BWPD from the Frio sand. The well was equipped with 1-inch tubing containing gas lift valves inside 2½-inch J55 production tubing. The 1-inch tubing extended to a depth of 2001 feet; total depth of the well was 5547 feet. A packer was set at 5482 feet. At 2468 feet, the 2½-inch tubing was swaged to 2-inch tubing. Severe corrosion was found to be at the swage, 467 feet below the bottom of the 1-inch tubing. It was doubtful that inhibitor applied in the conventional manner would reach even the bottom of the 1-inch tubing.

Corban was then pumped into the formation. Ten gallons of concentrated Corban 101 were mixed with 12½ barrels of salt water. Since this one treatment, water produced by the well has been analyzed several times. The following table shows the results.

Date	Iron, Parts Per Million	Remarks
4-6-56	132	Just prior to described treatment
4-11-56	41	First test after treatment
4-17-56	80	Second test after treatment
4-25-56	55	Third test after treatment
5-14-56	27	Fourth test after treatment
6-6-56	23	Fifth test after treatment Iron content reduced 83% at this time
9-1-56	37	Sixth test after treatment Iron content still reduced by 72% compared to that before treatment

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TECHNICAL TOPICS

Improved Fabrication Techniques and Lower Cost Favor Titanium's Use*

Abstract

Fabrication characteristics of titanium are discussed in connection with the proposed use of the metal for chemical equipment. Principal physical properties of two grades of commercially pure titanium are listed and a comparison made with Type 304 stainless steel. Cost data are given to show the economic feasibility of using titanium for a reactor and heat exchanger as contrasted with that of two other metals.

A number of recommendations are made regarding the procedure to be followed in welding titanium.

Introduction

TITANIUM has progressed in a relatively short time from a laboratory curiosity to a material of construction readily available for corrosion resistant applications. While the present price per pound of titanium prejudices many potential users against it (for example it is 18 times more expensive than Type 316 stainless steel) it is much lighter than stainless steel and when special fabricating techniques, such as liner type construction are used, finished titanium equipment may cost only five times as much as Type 316. In applications where the service life of titanium is four or five times that of stainless steel, titanium well may be the most economical material to use. Even at its present price it behooves the corrosion engineer to consider titanium for severe corrosion service.

Physical and Mechanical Properties

Three commercially pure grades of titanium are AMS 4902, 4900 and 4901. While there are a number of titanium alloys with greater strength and corrosion resistance equivalent to the commercially pure grades, in general only the commercially pure grades have been used for corrosion resistant applications. Some important physical and mechanical properties of the two most common commercially pure grades of titanium are shown and compared with Type 304 stainless steel in Table 1.

While tensile strength of titanium is about the same as that of stainless steel, its yield strength is considerably higher. In designing titanium equipment to the American Society of Mechanical Engineers Boiler Code the lowest value is used of

1. ¼ of the tensile strength
2. ⅔ of the yield strength
3. The stress producing a secondary creep rate of 0.1 CUR (creep rupture units) or
4. ⅘ of the stress producing rupture in 100,000 hours.

This results in allowable stress values for titanium that are slightly lower than that of stainless steel for AMS 4902 and higher for AMS 4901. Density of titanium is 57 percent that of stainless steel important in aircraft applications and an important volumetric advantage when it is used for corrosion resistant equipment.

Thermal conductivity is only slightly less than that of stainless steel and is higher than Hastelloy Alloy C. For medium and low rates of heat transfer, these differences of thermal conductivity are of little importance in the design of heat exchangers. Thermal expansion is considerably lower than that of the 300 series stainless steels, which is important in designs where dissimilar metals are used. Because of the low thermal expansion the 400 series stainless steels often are the best choice for titanium liner construction. Tensile modulus of elasticity is about one-half that of stainless and carbon steel. This property may be a disadvantage in using light sections of titanium without a backing where rigidity is a consideration, forcing use of heavier thicknesses than would be the case with stainless steel or other ferrous alloys.

Titanium, like stainless steel, owes its corrosion resistance to the formation and maintenance of a protective, passive film, presumably of a stable oxide. The film on titanium, more stable than its counterpart on stainless steel, does not break down readily in environments such as chlorides. Titanium does not pit in chloride environments like stainless steel.

Those interested in some of the data on titanium corrosion resistance are invited to inspect the references[1, 2, 3] listed at the end of this article. An account of a disastrous explosion that occurred when commercially pure titanium and titanium-manganese alloys were tested in fuming nitric acid has been published.[4] Red fuming nitric acid is also the only environment found to date that causes stress corrosion cracking. This can be avoided by stress relieving at 1000 F for one-half hour.

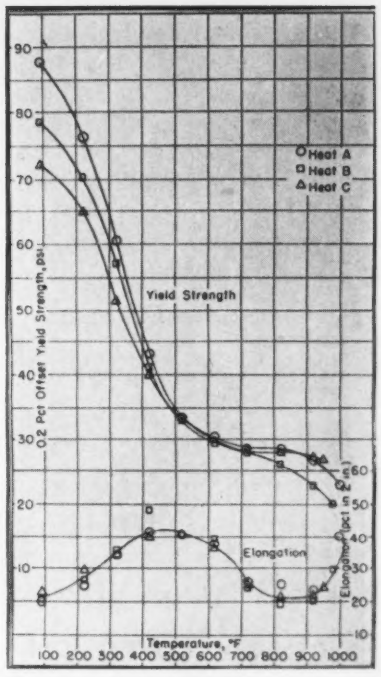

Figure 1—Data illustrating advantage of working titanium in the heat range 500-600 F.[5]

Titanium is resistant to dilute solutions of hydrochloric and sulfuric acid at room temperature. Addition of oxidizing agents such as cupric ions, potassium dichromate, nitric and chromic acids greatly improve titanium's corrosion resistance in hydrochloric and sulfuric acid. A striking illustration is titanium's almost complete resistance to aqua regia at room temperature.

Forms Available

Titanium is available in a wide range of wrought forms including plate, sheet, strip, tubing, bars, wires, forgings, etc. Maximum size restrictions of the various forms are generous considering the infancy of the metal. The maximum size of some plates and forgings are limited to slightly over 1000 lb. Tubing (welded, welded and drawn, and seamless) is

(Continued on Page 120)

TABLE 1—Properties of Commercially Pure Titanium vs Type 304 Stainless Steel

	Aeronautical & Material Specification, Ti						Type 304
	AMS 4902 psi Thousands		AMS 4900 psi Thousands		AMS 4901 psi Thousands		PSI Thousands
PROPERTY	Min.	Max.	Min.	Max.	Min.	Max.	Minimum
Ultimate Tensile Strength	50		65		80		75
Yield Strength 0.2% Offset PSI	40	65	55	80	70	95	30
% Elongation in 2 Inches	20		18		15		30

★A paper presented under the title "Fabrication of Titanium" by Richard E. Avery and S. C. Orr, Pfaudler Co., Rochester, N. Y. at a meeting of Northeast Region, National Association of Corrosion Engineers, Philadelphia, Pa., October 15-17, 1956.

Improved Fabrication—

(Continued From Page 119)

available from titanium producers and a number of mills.

Titanium differs from most alloys and metals in that it is so difficult to cast that castings are not readily available commercially.

Sponge material with lower carbon, nitrogen and oxygen, titanium obtained today is of much higher quality than it was a few years ago. The reduction of these elements has produced a more ductile material and one that can be welded without excessive danger of contamination. Double melting also has helped to produce more homogeneous material.

Spring Back is Characteristic

A striking characteristic of titanium is its spring back when cold formed. Fifty or 60 degrees spring back is not uncommon in some operations. The degree of spring back is determined by the yield point and the yield point can vary considerably in different heats and lots. Spring back can be largely eliminated by warm working between 500 to 600 F. Figure 1 illustrates the advantage of forming the metal in this temperature range.[5] Note that the three heats have a spread of 15,000 psi in their yield strength at room temperature. If bent through an angle of 90 degrees over a 1-inch mandril, 0.025-inch sheet from the three lots would spring back between 50 and 60 degrees. When heated to 450-500 F yield strengths are nearly the same and the same amount of spring back from each would be expected. An improvement in ductility also is obtained in the range of 450-600 F.

Tank heads have been fabricated by sandwiching the titanium sheet between two pieces of clean, mild steel and heating in the furnace to approximately 1000 F and forming the sandwich complete by pressing. The heating was not high enough to cause oxidation or embrittlement of the titanium and good quality heads were produced. This is a good illustration of the amount of forming that can be done if proper preheat temperatures are used.

Titanium's particular characteristics require different machining techniques but when these characteristics are recognized parts can be machined on a routine production basis. Titanium may be machined on standard equipment with high-speed, cast alloy or carbide tools.

Welding Characteristics and Techniques

Four interstitial alloying elements, oxygen, carbon, nitrogen and hydrogen have a very pronounced effect on the mechanical properties of commercially pure titanium. It is for this reason that the melting is done in vacuum inert atmosphere furnaces. The United States Air Force Specification Bulletin No. 108A places the following limits on wrought alloys[6]: Oxygen, 0.20% max.; nitrogen, 0.27%; carbon, 0.20%. In weldments, the presence of these amounts of interstitials would result in low ductility and poor impact characteristics. It is the tendency of titanium to absorb these elements, either in the molten weld metal or in the heat affected zone, that largely dictates the welding processes that are successful and the modification of each process that must be employed to minimize contamination. This immediately rules out oxyacetylene and arc welding. Titanium has been welded successfully with the inert-gas-shielded metal arc process, spot, seam and flash welding.

Spot, seam and flash welding of commercially pure titanium requires techniques that are not much different from those used on some of the more common materials of construction. They can be welded without special gas shielding but there have been some cases where inert gas shielding was found beneficial in flash welding. Discussion will be limited to the non-consumable electrode inert-gas shielded-arc process. The consumable electrode process also has been used successfully but since this process is used mainly in heavier gauges it has not found wide use.

Proper Cleaning Required

Before welding titanium, proper cleaning is necessary. The cleaning practice is dictated largely by the surface condition. If it is free from scale, such as sheet received from the mill, degreasing is sufficient. It is good practice to use an abrasive cloth wheel to clean the weld joint area before welding. If there is a light scale on the surface, from stress relieving, for example, the surface should be acid pickled. A very satisfactory solution is one containing 20 percent or more nitric acid[1] and 2% to 4% hydrofluoric acid. Pickling times range from one to twenty minutes and bath temperatures range from 80 to 160 F. If heavy scale is present it is best removed by grit or sand blasting, followed by the nitric-hydrofluoric acid pickle. Molten salt baths[2] have also been used to remove heavy scale but with some there is a danger of hydrogen pick-up.

Joint designs for titanium welding are essentially the same as those used on other metals. Before welding, the burrs should be removed from sheared edges, usually with a draw file. Because molten weld metal has a higher fluidity than, for example, that of stainless steel, it is advisable to weld as close to the flat position as possible.

Both helium and argon, and in some cases a mixture of the two, have been used for shielding gases. Helium has the advantage of higher welding speeds and better penetration, while argon offers greater arc stability. On a whole, argon has been used more extensively, particularly in manual welding.

In the early days of arc welding titanium, inert gas chambers were used extensively. The three basic types were,

1. A construction such that a vacuum was first drawn in the chamber which then was filled with inert gas,
2. A type that was purged with inert gas until the atmosphere was essentially free from air,

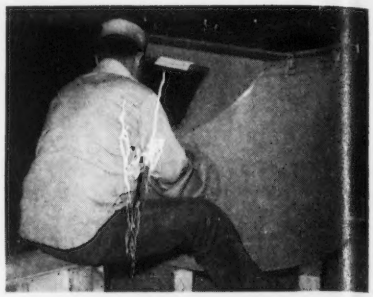

Figure 2—Purge chamber used in welding small titanium parts.

Figure 3——One type of trailing chamber used in welding titanium in inert atmosphere.

(1) Picking solutions with less than 20% by weight nitric acid may cause absorption of hydrogen.

(2) Hydrogen embrittlement may be caused by improper use of molten salt baths.

(Continued on Page 122)

TECHNICAL REPORTS

on

GENERAL CORROSION PROBLEMS

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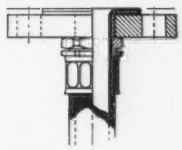

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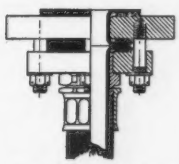

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Improved Fabrication–

(Continued From Page 12)

3. A collapsible type that was back-filled with inert gas after collapsing, to remove air.

Figure 2 shows a purging unit used in Pfaudler's plant. After placing all equipment in the chamber, it is purged with argon until the volume has been changed five times and Orset analysis of the chamber atmosphere has detected no oxygen. During welding the gas flow is maintained in the torch so that most of the molten metal is protected by the gas from the torch and the chamber gas protects the heat-affected zone and the cooling weld metal.

Welding Equipment Improved

The inert chamber method has been losing favor in production work, a principal reason being the improved quality of sponge. Most titanium produced today can tolerate a small pick-up of the interstitial elements without detrimental effect.

Improved back ups to protect the underside of the weld and baffles and trailing shields to hold the gas around the top of the weld have been devised. An illustration of one type of trailing shield is shown in Figure 3. These are important advances because it obviously would be impractical to weld large sized vessels in enclosed chambers. However, inert gas chambers still have their uses and often are the easiest way of welding small complicated shapes that do not lend themselves to backups, baffles and trailing shields.

A number of destructive tests are useful in evaluating titanium welds. Most common are tension, bend, hardness and notch-toughness tests. Tension test specimens may be of the flat bar type with the weld longitudinal or transverse to the direction of force, or the round bar specimen that normally includes the weld metal only. Because thick specimens with a number of passes are needed for the round bar specimens they are not used as often as the flat bar specimens. Flat bar specimens with the weld transverse give a good measure of the joint efficiency, but failure usually takes place in the base metal. The flat bar specimens with the weld longtitudinal give a good measure of the weld joint ductility and a better indication of the weld joint tensile strength. The latter has not been used as extensively as the transverse specimen but its use has been strongly recommended by some investigators.

TABLE 2—Cost of 200-Gallon Reactor: Comparison of Three Materials of Construction

	Titanium Lined	Type 316	Hastelloy Alloy B
Average cost per lb.	$ 14.60	$ .8025	$ 2.62
Material required, lb.	250	785	675
Material costs	$ 3,650.00	$ 630.00	$1,768.00
Cost of finished reactor	$12,150.00	$2,391.00	$4,670.00
Ratio to stainless	5.19	1.0	1.95

TABLE 3—Cost of Model 1007 HX 104 Sq. Ft. Fixed Tube Heat Exchanger: Comparison of Three Materials

Type Material	Sales Price	Cost/ Sq. Ft.	Ratio to Stainless
Type 316	$1575	$15.15	1.0
Hastelloy C	$4330	$41.60	2.75
Titanium	$7250	$69.60	4.60

Two Types of Bend Tests Used

Two types of bend tests commonly used are the guided bend or the free bend, in each of which the weld may be longitudinal or transverse to the direction of stress. There has been no industry-wide agreement to standardize on one type of test, or the acceptable limits for each. The guided, 105 degree bend test has proved a good test for welds in material up to about 10 gauge. In heavier materials Pfaudler favors the free bend test, used primarily as a quality control check on the welding procedure and performed before each new piece of equipment is built or when a different type of weld is made.

Non-Destructive Test Methods

The three most commonly used nondestructive tests are X-ray, dye-penetrant and visual inspection. X-ray is useful in checking for porosity, weld penetration and generally will detect cracks. Dye-penetrant inspection is useful in detecting cracks and is often a standard inspection for welds that are made under restraint conditions. Welds usually are examined visually to check the gas protection during welding. A heavy scale indicates that the weld has been severely contaminated by the atmosphere and has poor ductility. The surface of the weld may be shiny but have a range of colors from grayish-blue to a violet-brown. These colors result from an oxide formation when the metal is cooling after solidification.

However, much more important than the interstitial pick up received when the solidified metal cools is the pick up in the molten metal. Molten titanium can rapidly absorb large quantities of the interstitial elements while the rate of diffusion in hot solid metal is much slower. Consequently, a discolored weld could have had excellent protection when molten but inadequate protection during cooling and still have acceptable physical properties. The reverse also could happen, that is, a weld could have had poor shielding when molten and good protection during cooling. This weld would be bright and shiny but would have very poor mechanical properties. There is definitely a need for a good nondestructive test for weld metal contamination.

Some investigators have found the circular patch weld restraint tests useful in studying the capacity of welds to withstand high shrinkage stresses or slow sustained strain-aging stresses. Some experience to date indicates that tests of this nature are desirable and necessary.

Cost of Titanium Equipment

Although on a weight comparison, titanium is much more costly than most other corrosion resistant materials, the cost of finished equipment is more favorable to titanium, and when it is considered that further cost reductions are in immediate prospect, it becomes even more interesting for future equipment.

It is necessary also, in comparing the prices of engineering materials to weigh the factors involved in capital amortization, which may favor titanium in some cases because of its extended service life when compared to other less costly materials. Such factors as down time, lost production and maintenance also need to be taken into account.

Some of these points are illustrated in Tables 2 and 3. For simplicity, the cost per pound of the materials are average prices figured by dividing the weight of material required by the total cost of the material. The cost per pound of all the materials varies widely depending on the size, quantity, etc. The cost of the carbon steel used for backing the titanium liner is included in the cost of the finished reactor and is not shown separately. In this small heat exchanger the price comparison is even more favorable for titanium than in the 200 gallon reactor shown in Table 2. This is largely because of a liner type construction on the tube sheets and bonnets.

Conclusion

It is illustrated that in the designs considered titanium equipment is about five times as expensive as Type 316 stainless steel. This factor will vary depending upon the design, size and other considerations. An attempt has been made also to illustrate that titanium equipment for the chemical industry is a reality and should receive the corrosion engineer's consideration for severe corrosion services.

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6. Welding of Titanium and Titanium Alloys, TML Report No. 31, February 7, 1956.

HOW CORROSION IS INDEXED

1. Alphabetical subject and author indexes are published in December at the end of the calendar year volume of 12 issues.
2. Abstracts of articles appear in the Corrosion Abstracts section of CORROSION.
3. Abstracts of articles appear in the NACE Bibliographic Surveys of Corrosion.
4. Abstracts of articles appear in the Corrosion Abstract Punch Card Service.
5. CORROSION is Indexed regularly by Engineering Index.
6. CORROSION will be indexed, beginning in January 1958 by Applied Science and Technology Index.
7. A 10-year alphabetical subject and author index (1945-54) has been published and is available for sale.

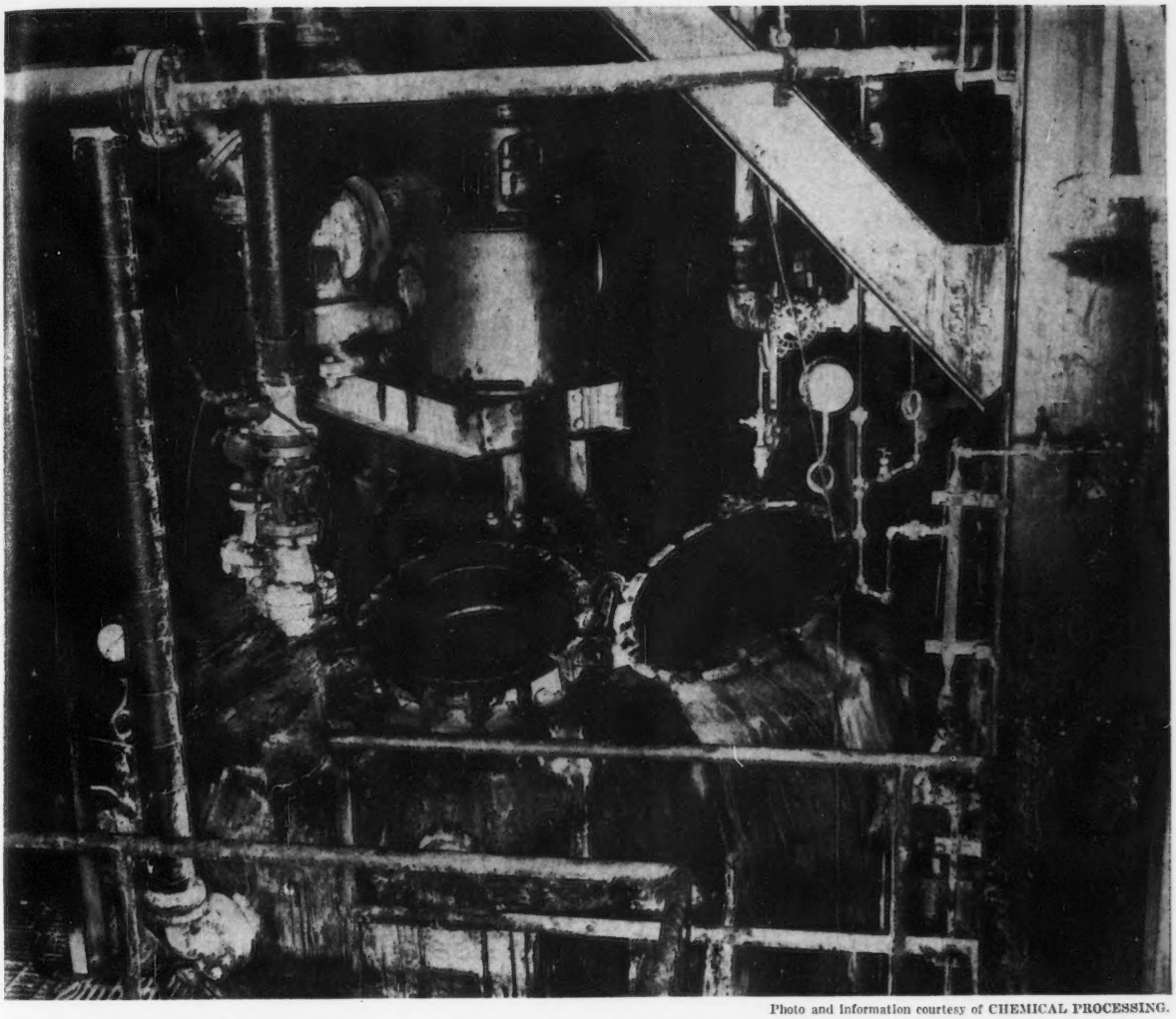

Photo and information courtesy of CHEMICAL PROCESSING.

Corrosion protection of Monel nickel-copper alloy extends over manway lip in this Monel-clad steel-jacketed autoclave. Solid Monel alloy is used for baffle plates and agitator parts. Three of these units were fabricated by the Colonial Iron Works Co., Cleveland, for Westvaco Chlor-Alkali Division of Food Machinery & Chemical Corporation.

Now good for three times the life! Monel withstands attack by abrasive silica in 50% caustic

This Monel-clad autoclave has been producing sodium silicate for six years at Westvaco's plant in South Charleston, W. Va.

Inspection indicates that the unit is good for at least another four years. That's better than three times the life of its predecessor.

In-plant corrosion tests determined choice

As the basis for redesign of earlier equipment, engineers at the Westvaco plant relied on the results of in-plant corrosion tests conducted cooperatively with Inco's Development and Research Division.

These tests indicated that Monel* nickel-copper alloy provided the maximum resistance to the abrasive-corrosive action of silica flour in an agitated 50% caustic at 175 psi and 350°F. The tests further indicated that a 0.125-inch Monel alloy cladding on steel could be expected to last ten years or more.

The first few months experience with the initial Monel-clad unit confirmed test results, so a second, and later a third, unit was installed. Maintenance and replacement savings have been substantial and all three units have plenty of service life left.

Metals that fail too soon call for corrective study and action

Inco is prepared to help you determine the nature and causes of metal failure and suggest materials able to withstand the conditions. All it takes to get this help is a note to our Development and Research Division.

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Variables Affecting Validity Of Accelerated Coating Tests*

Abstract

Some of the variables that affect the performance of coatings that need to be considered in evaluating results of accelerated tests are given. These include variations in materials, formulation procedure, surface preparation and the materials used for surface preparation, environmental factors, application variables. 2.3.2

MANY CORROSION engineers are still dubious about the effectiveness of surface coatings for controlling severe corrosion. In many cases their doubts are well-founded, because there is certainly no single, miracle working coating which will automatically handle all problems. There are, however, several types of coatings now available which perform effectively even under the most severe conditions, provided the proper coating is selected and carefully applied. The big problem is how to select the right coating; and unfortunately there is no simple answer.

Obviously it would be desirable to have some kind of chart or catalog which would make it possible for the corrosion engineer to select the best coating for a given set of conditions. National Association of Corrosion Engineers technical committees have made great strides in this direction, but most of the committees have discovered that it is impossible to draw up a chart which will infallibly indicate the one best coating for the job at hand. There are too many variables in conditions and in coating materials and formulations.

For example, there are today more than a dozen commercially available vinyl chloride type resins which can be used for surface coatings. These resins are by no means the same. They differ widely in chemical resistance, weather resistance, water resistance, ease of application, adhesion and in other less obvious ways. In addition to variations in properties there are also variations from plasticizers, pigments, stabilizers and solvents. Manufacturing methods also have a surprisingly great effect upon the properties of a coating. Often the manufacturer's skill in formulating and processing influences the coating performance more than the choice of resin.

Order of Mixing Important

Figure 1 demonstrates the importance of manufacturing "know how." Two test rods illustrated were exposed to hydrochloric acid. The coating on each specimen was prepared according to exactly the same formula, using standard manufacturing processes, the difference being the order in which some of the ingredients were mixed. This made the difference between success and failure of the coating in the environment and shows the futility of attempting to evaluate a coating on the basis of resin type or formula alone.

It is true that a review of resin properties can assist in guiding an investigation. However, the one best material for any set of conditions can be determined by testing only.

Obviously the best method would be to test under actual operating conditions. Because it seldom is practical to test a coating by actually lining a tank, some type of laboratory test is required to give an indication of the suitability of a coating. Preferably, some accelerated method should be found so results can be obtained rapidly.

Hazards of Accelerated Tests

Unfortunately, many corrosion engineers have found that accelerated tests do not indicate accurately performance obtained in actual use. The principal cause of the discrepancy is failure to take into account all variables in factors which affect coating performance. These principal factors are: 1. The environment. 2. The type of surface and its preparation. 3. Care and uniformity of application.

It is obvious to anyone familiar with protective coatings that these factors are important, but even the most expert sometimes have been surprised by the degree of variation found under actual operating conditions. Reliable tests must take these factors into consideration.

A few examples follow, showing what type of variation in the influencing factors can be expected and how failure to recognize them can result in invalid tests or coating failure.

Influence of Environment

Obviously, the coating must be resistant to the environment, which, in the case of a tank lining, is the contents of the tank. Testing the resistance of the coating itself ordinarily is one of the most straightforward problems. It is always possible to test the coating by applying it to small specimens and exposing them to the material to be held in the tank.

It is not always so simple, however, to accelerate test results without introducing other factors not expected in service. For example, if a coating is to be used to contain dilute acid it would be logical, to some degree, to accelerate the test by increasing the concentration of the acid or the temperature. However, it is possible that either one of these could lead to confusing or misleading results. For example, some coatings will hold up better in concentrated sulfuric acid than they will in a dilute solution. Thus, if the exposure is to a 5 percent solution the wrong answer would be obtained if the materials were tested in a solution of 50 percent concentration. If an increase in temperature is used to accelerate the test, results could be thrown off again because some coatings are affected to a greater degree than others by increased temperature. A test at elevated temperatures will not necessarily indicate the comparative resistance of materials at room temperature.

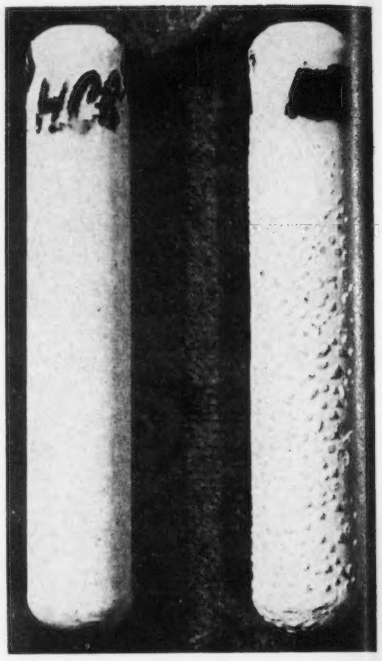

Figure 1—Specimens with identical coatings after exposure to hydrochloric acid showing the results of mixing ingredients in different order.

When chemical resistance tests are made, the results should be evaluated two ways: 1. The effect of the environment on the coating, and 2. The protective value of the coating. These are not necessarily the same, because a coating can be apparently unchanged and yet allow extensive underfilm corrosion, which would mean eventual failure.

Changes in physical properties of the coating also can occur and these are sometimes harmful, sometimes beneficial. Some coatings actually are improved by exposure to acid or alkali, as the case may be and their physical properties should be evaluated after chemical test.

Another pitfall is failure to take into account the result of environment changes. For example, suppose a lining is required for a tank which will contain alternately caustic soda and ethylene dichloride. To begin with, immersion tests are made in both liquids and a coating is found which is satisfactory in either. It might be concluded from these tests that the coating would be suitable for handling the alternate ladings. However, laboratory tests in which the coating is exposed alternately to caustic and ethylene dichloride, two days in each, show signs of failure after four cycles. Multiple ladings frequently result in this type of failure and tests of tank linings for this purpose should simulate the exact conditions as closely as possible, including alternate exposure to the various cargoes at the actual operating temperatures.

Type of Surface and Its Preparation

It is extremely important to know the exact type of surface over which the coating is to be applied. Most tanks encountered in the transportation field are made of mild steel, but occasionally a

(Continued on Page 130)

★A paper presented under the title "Accelerated Testing of Protective Coatings," by John I. Richardson, Technical Director, The Amercoat Corp., South Gate, Cal. at a meeting of South Central Region, National Association of Corrosion Engineers, San Antonio, Texas, October 23-26, 1956.

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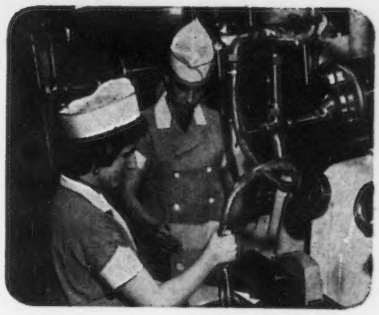
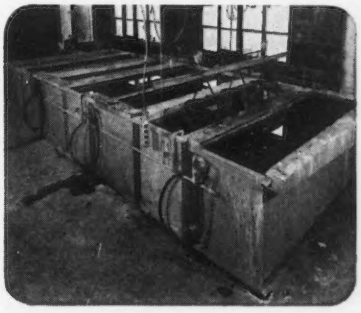
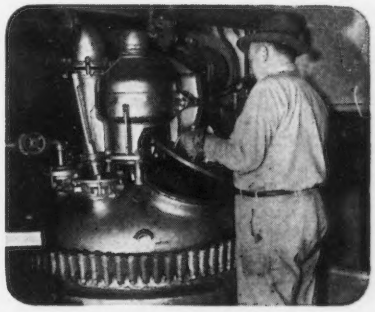


AS TUBING OR HOSE to pipe taste-sensitive liquid foods or corrosive chemicals. Tygon Tubing is glass-clear, flexible as a piece of string, resistant to acids and alkalies, non-toxic, sterilizable, and is made in bores from 1/16" to 2".

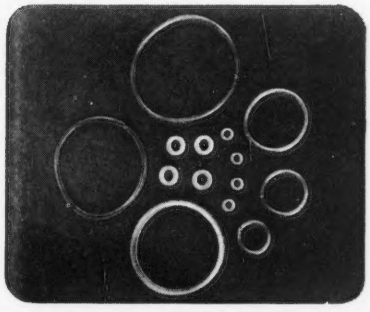
AS A PAINT to protect plant and equipment from attack by corrosive fumes and gases. Applied by brush or spray, Tygon air dries quickly to form a tough impermeable plastic skin that shrugs off acids, alkalies, oils, water and alcohols.



AS A HEAVY-DUTY LINING to protect the interior of pickling, plating, chemical processing and storage tanks from destruction by corrosive solutions. Easier to install than rubber linings, Tygon can be applied to tanks of any size or shape.



AS A CORROSION-RESISTANT GASKETING. Tygon shows no chemical deterioration with age. Gaskets remain flexible and tight, unaffected by weather or exposure to chemicals. Tygon gaskets are available in an almost unlimited size range.



AS MOLDED ITEMS. Tygon's toughness, durability and flexibility, coupled with staunch chemical resistance, offer pronounced performance characteristics for grommets, washers and molded mechanical goods items in virtually any shape.

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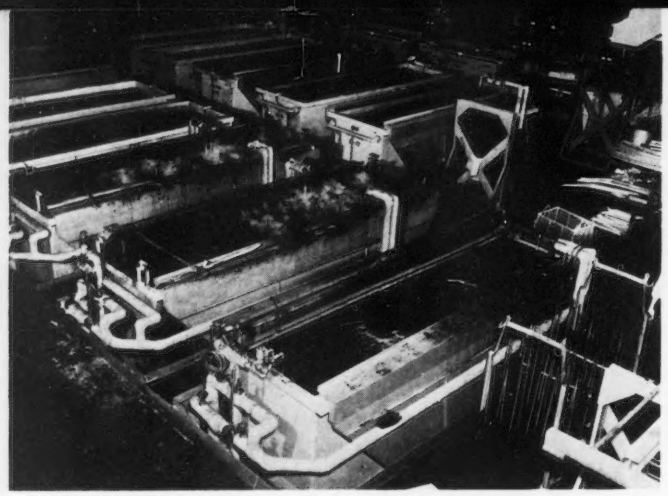

Figure 1—Battery of metal treating tanks in an aircraft plant lined with plastisols.

Using VINYL PLASTISOLS for Resistance to Chemicals and Abrasion★

Abstract

Characteristics of vinyl plastisols are described. Examples of their successful use are given when applied as coatings on plating racks, bubble trays and duct systems, as linings on tanks containing corrosive liquids, as linings for centrifugal cleaners in the pulp and paper industry and as reusable drum linings. Their resistance to mechanical abuse is emphasized.
5.4.1

THIS PAPER covers vinyl compositions which can be applied to intricate and complex shapes as readily as vinyl paints but at thicknesses approaching that obtainable with sheet linings. These compositions are called plastisols. Plastisols are fluids, so they displace rather than pocket air, contain little or no solvent, hence there is no risk of solvent entrapment and they can be formulated to cover edges and sharp projections to form seamless, tough and resilient films or linings.

Vinyl solution coatings or paints are prepared by dissolving vinyl resins in solvents, usually ketones or mixtures of ketones and aromatic hydrocarbons. Resin content generally is limited to 15 to 30 percent of the total weight due to its limited solubility. After application the solvent evaporates from the fluid film leaving behind the solids content of the coating, the bulk of which is resin and pigment. Even with heavy pigment loading, film thicknesses seldom exceed 10 to 15 mils.

Characteristics of Plastisols

Plastisols are dispersions or suspensions of discrete vinyl resin particles in nonvolatile liquids known as plasticizers. Transformation of this dispersion into a continuous film is governed by the solvating characteristics of plasticizers on the vinyl resin. Solvent action of plasticizers on the vinyl resin is a function of temperature. At normal room temperature the plasticizer has very little solvating effect but it increases until a temperature is reached at which the solvency power of the plasticizer is sufficient to completely dissolve the resin.

A plastisol coating will remain in its original liquid condition indefinitely but when heat is applied solvation of the resin in the plastisol creates a gel-like structure. For best film properties, the coating must reach a temperature at which the resin completely dissolves in the plasticizer, at which time the coating changes from a two phase dispersion to a single phase continuous film. The temperature at which complete solution occurs depends upon the plasticizer or plasticizer blend, almost always between 350 and 375 F. The cooled plastisol film becomes a tough, resilient, chemically resistant plastic.

Because plastisols contain no volatile solvent and because transformation to continuous films does not depend on release of volatile reaction products it is possible to dip, spray, or mold films ranging in thickness from 0.001 to 0.25-inch. Plastisols have no specific adhesion to metal, so for adequate bonding special primer systems are required. These primers are based on mixtures of vinyls, phenolics, butadiene acrylonitriles and epoxies. A properly formulated primer-plastisol system usually averages a bond strength of 50 to 100 pounds per inch. Some primer systems are exceptionally resistant to corrosion undercutting in the event scoring of the plastisol coating occurs.

Applications Are Numerous

Possibly the first commercial use of plastisols on metal was as insulation for electroplating racks. In this application they have replaced all vinyl solution coatings and rubber insulation almost completely. To operate successfully as a rack coating, the plastisol not only must confine the current to the work being plated but must be chemically resistant as well. Typical solutions encountered in electroplating are given in Table 1.

Their toughness and abrasion resistance is demonstrated by their use as rack coatings where, prior to plastisols, solution rack coatings formulated from high molecular weight vinyls had necessary chemical resistance but failed within a few months because of their inability to withstand mechanical abuse. In contrast to this, plastisol rack coatings remain in automotive plant service without failure throughout the useful life of racks. Because of design changes racks usually are obsolete or require major alterations every two years. In other industries where changes in rack design are less frequent plastisol rack coatings withstand three to four years of daily use.

Hot Dip Technique Used

In applying plastisols to plating racks, a hot dip technique is used which also is applicable to many pieces of chemical equipment such as agitators, fan blades, filter plates, bubble cap plates and even to small tanks. Advantage is taken of the increased solvating effect of the plasticizer on the resin with increased temperature.

To apply a heavy thickness, the article to be coated is preheated and while still hot is immersed in the plastisol. The heat given off by the immersed hot article will set or gel the plastisol adjacent to the article. This gelled coating will not drain off when the article is withdrawn. The coating then is fused by baking. By this method it is possible to apply coatings ⅛ to ¼-inch thick in a single dip.

While this technique is practical for handling comparatively small articles and is employed even in the slush coating of small tanks, a spray technique is necessary for large equipment. The plastisol used in hot dipping drains freely. Although it can be spray applied, preheating of the article being sprayed is required to obtain a heavy build. Often this is not practical, so for spray application a plastisol must have a fine balance of flow characteristics. Too much flow results in sags, runs and lack of film build while sufficient flow is necessary to fill voids in the metal

TABLE 1—Typical Solutions Encountered in Plating Baths

Water Rinses—Temperatures ranging from ambient to boiling.

Detergent Cleaners—8 oz. per gal., temperatures from ambient to boiling.

Alkali Cleaners—16 oz. caustic per gal., temperatures from ambient to boiling.

Chromium Plating Bath—34 oz. chromic acid per gal., temperature 100 F to 150 F.

Nickel Plating Bath—40 oz. nickel chloride per gal. at 150 F to 160 F.

Copper Plating Bath—3 to 4 oz. of both copper and sodium cyanide per gal. at 140 F to 180 F.

(Continued on Page 128)

★A paper presented under the title "Plastisols Combine Thickness With Chemical Resistance," by F. L. Scott and W. C. Hosford, Metal and Thermit Corp., Rahway, N. J. at the 13th Annual Conference, National Association of Corrosion Engineers, St. Louis, Mo., March 11-15, 1957.

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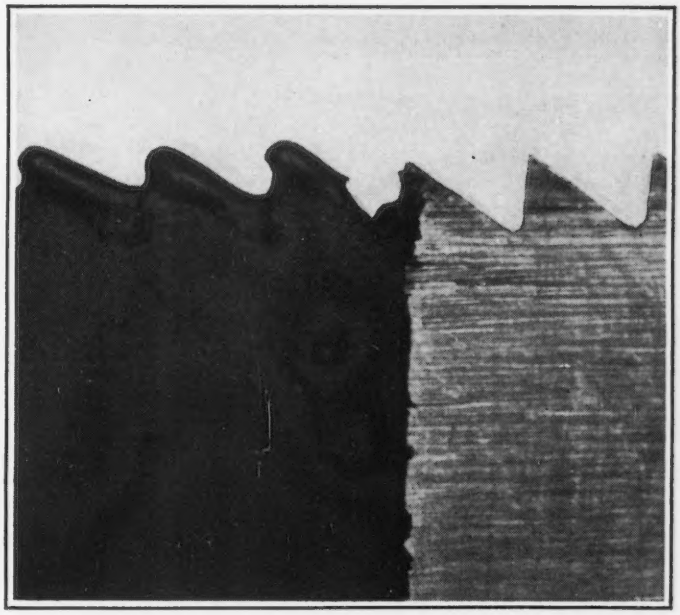

The unique ability of Prufcoat Hot-Spray Vinyl to provide the essential extra protection needed on sharp edges — and other corrosion-vulnerable trouble spots — is clearly documented by this photo showing sharp saw teeth at left heavily vinyl coated.

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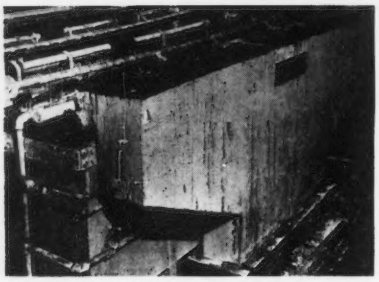

Figure 2—This unit is shown after a year's service after coating with a standard maintenance paint.

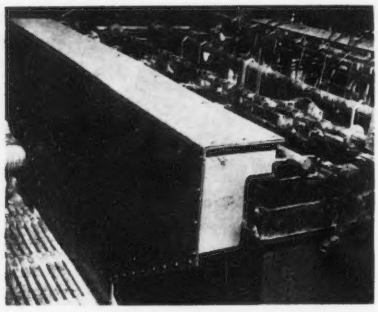

Figure 3—This unit, protected by a plastisol coating, also has had a year's service.

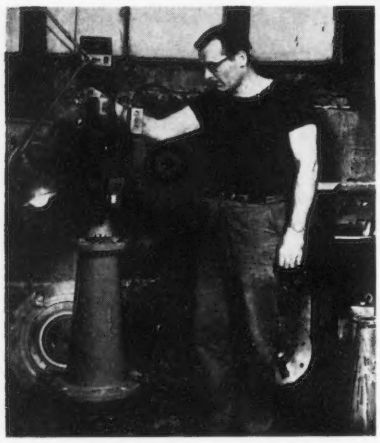

Figure 4—Grinding to close tolerance for flange fitting is another asset of plastisol linings.

TABLE 2—Effect of Corrosive Solutions on Plastisol Linings in Tanks (Two Years' Exposure)

SOLUTION	Concentration	Temp. Degrees F	Comments
Acetic Acid	25 oz/gal + 7 oz/gal $NaNO_2$	75	Blistered 6-9 Mos.
Chromic Acid	24 oz/gal	140	No Effect
Chromic Acid	33 oz/gal (Electrolytic)	131	No Effect
Chromic Acid	37 oz/gal	75	No Effect
Hydrofluoric Acid	15 percent	75	Blistered 9-12 Mos.
Hydro Fluorosilicic Acid	2 percent	75	No Effect
Hydrochloric Acid	18 percent	75	No Effect
Sulfuric Acid	10 percent	75	No Effect
Nitric Acid	8% + 2% H_2SO_4	75	No Effect
Cold Water Rinse	.....	75	No Effect
Hot Water Rinse	.....	160	No Effect
Copper Cyanide	3 oz/gal + 4.5 oz/gal NaCN 2 oz/gal $NaCO_3$ (Electrolytic)	140	No Effect
Ferrous Sulfate	7% + 2% HF	75	No Effect

Vinyl Plastisols—

(Continued From Page 126)

substrate, prevent the formation of air pockets and deposit a smooth, uniform film.

An early application of spray plastisols was as a lining for the exhaust system for a large electroplating plant where fumes consisted of mixtures of hydrochloric, nitric and sulfuric acids. A two-coat application of plastisol totalling 35 mils was employed. This duct system shows no evidence of deterioration after four years of service.

Bubble Tray Is Coated

Plastisols were used to coat a 5-foot diameter 400-pound bubble tray at the bottom of a 60-foot scrubbing tower, supporting a packing composed of carbon or ceramic rings. Gases containing hydrochloric and nitric acid fumes bubble through the packing while caustic solutions trickle down, removing and neutralizing the acid fumes. Sheet rubber previously used to protect these trays was adandoned in favor of plastisol because of their high cost. After two years the plastisol coated bubble trays are still standing up.

Successful application as exhaust system linings led to use of spray plastisols for tank linings.

Figure 1 illustrates a battery of tanks in the metal treating department of a major aircraft plant where the dichromate treatment of magnesium, the Alodine treatment of aluminum and copper-nickel-chromium plating are performed. Fifty-two tanks in this department are lined with spray plastisol. Table 2 lists some of the solutions in the plastisol lined tanks. Large tanks frequently are fabricated and coated in sections and bolted together after the lining has been baked.

At the nearby engine division of this company where jet and piston engines are built, spray plastisols, in addition to being used as linings for rinse and plating tanks, duct work, fans and chemical filters, are used at 15 to 20 mils thickness as the exterior maintenance coating on over 1000 tanks.

Plating Tank Bottom Protected

In plating installations overflow commonly is discharged into drainage troughs beneath the tanks where continuous exposure to acid fumes causes rapid corrosion of bottoms. The thickness and greater resistance to mechanical abuse of the plastisol coating gives good protection in these inaccessible areas. Figures 2 and 3 show how a conventional maintenance paint compares with a plastisol coating after a year of service. Plastisol tank lining often is applied in four to five spray coats to build a thickness of 80 to 100 mils but plastisol formulations recently developed will build 80 to 100 mils in two spray coats. Different color plastisols are used in alternate coats as an aid to proper application. Spot checks of the wet film thickness are made using a wet film thickness gage. After curing, the thickness is gauged again and tested for pin holes with a high frequency spark tester.

Many materials of construction have been investigated for the fabrication of centrifugal cleaners developed to remove foreign matter from pulp and paper stock. All metal cleaners were unsatisfactory because of excessive wear. An extreme example of the wear which can occur in these cleaners is illustrated by the fact that a metal particle, having about the same mass as a ½-inch bolt, travelling around inside the cleaner in the same orbit can completely shear an unlined 5/8-inch thick cast iron cleaner in a few weeks. Four years ago one manufacturer adopted a 3/8-inch molded plastisol lining for its cleaners and today there are about 6000 plastisol lined cleaners in operation in over 400 pulp and paper plants throughout the United States, Canada and in foreign countries.

High Finish Is Retained

Figure 4 illustrates surface grinding of a flange gasket molded as an integral part of this lining. Ability to closely reproduce a highly polished mold surface and maintained the finish under extreme conditions of abrasion and under exposure to a variety of solutions accounts for the success of plastisols in this application.

Versatility of plastisols is demonstrated by their use as molded liners at thicknesses of 3/16 to 3/8-inch, as sprayed tank linings at 60 to 125 mils and as permanent maintenance finishes at approximately 20 mils. Phosphatized and primed steel containers lined with approximately 8 mils of plastisol fused at 365 F have a record of four to five-fold increase in the number of trips per drum. Plastisol drum linings overcome some of the difficulties with conventional coatings because they have excellent flexibility and resistance to impact and their toughness preserves film integrity against mechanical abuse while film thickness is sufficient to seal off seams and prevent corrosion contamination of product. Some materials successfully handled in drums so lined are concentrated phosphoric acid, magnesium fluosilicate (concrete hardener), detergent concentrates, 85 percent lactic acid and acid dyes.

DISCUSSIONS

Question by Albert K. Ackoff, Eastman Kodak Co., Rochester, N. Y.:
What test method is used for checking pin holes, discontinuities, etc.?

Reply by F. L. Scott and W. C. Hosford:
Reference is made to NACE Technical Committee Report, Publication 54-4, on "Application, Technology, Physical Characteristics, and Chemical Resistance of Polyvinyl Chlor-Acetates"[3] in which two methods of testing for discontinuities are described. One method uses a high frequency spark tester when the linings are 1/16-inch or thicker. For linings rang-

(Continued on Page 130)

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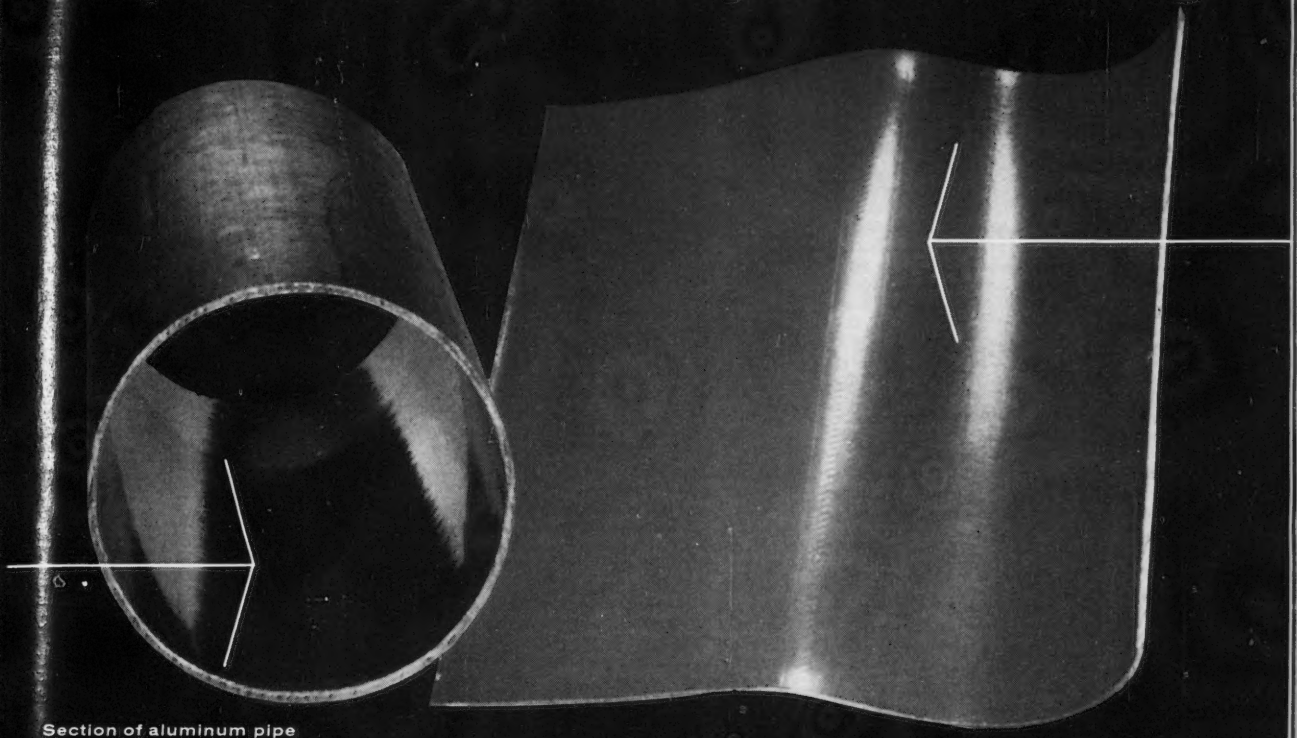
Unique TK-33 Baked Polyurethane Coating



Section of aluminum pipe coated with TK-33

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Variables Affecting—

(Continued From Page 124)

coating will be required for some other type of surface. Examples are stainless steel, aluminum, galvanizing, or other "metallized" surfaces to which zinc or aluminum has been applied by flame-spraying. A coating which is suitable for use over one of these materials will not necessarily be suitable for the others, so tests should be made over the type of surface actually to be encountered.

When evaluating surface preparation, the exact quality and degree of variation should be known. For example, blast-cleaning can be specified and the surface can be cleaned by this method in such a way that all rust, scale, and foreign matter is removed. However, the exact surface condition is not known unless the maximum profile depth is measured also. It has been previously pointed out by Arno Liebman[1] that the profile depth should be approximately one-quarter of the total coating thickness. Therefore, this should be specified and controlled in relation to the coating thickness for any given application.

Another possible source of difficulty is contamination after the surface has been blasted. This was observed during one of the first applications of a vinyl coating to a tank car a number of years ago. In this case the interior was blast-cleaned according to the specifications and the first coat was applied to the surface in the normal way. However, the next morning the prime coat was still tacky (and this was a material which normally dried hard within a few minutes). Air was circulated in the tank and another day of drying was allowed. The coating was still tacky.

Finally, an investigation of the history of the car revealed it previously had carried a load of beeswax. The sand-blasting had successfully removed the rust and scale but had also succeeded in spreading a thin invisible film of wax over the entire surface. Thus, surface preparation in this case was not satisfactory and the tank had to be steam-cleaned and reblasted.

Because many tank car repair shops use recirculating grit as an abrasive for blast-cleaning it is possible for a similar difficulty to occur whenever this system is used. The grit becomes contaminated after it has been used to clean out various materials which remain on the surfaces of cars and when reused, distributes these materials over every surface on which it impinges. In some cases the only way the surface can be cleaned thoroughly is to follow the grit blasting with another blast using clean sand. Though this problem is somewhat removed from that of accelerated testing of coatings, it is a variable which should be considered if the desired results are to be obtained from the coating application.

Application Factors Are Important

Another factor which must be considered is the continuity of the coating after it is applied. Under certain conditions some coatings will fail rapidly if any bare edges are exposed. An example of this is found when coatings are exposed to ammonium nitrate-ammonia. Many coatings will hold up successfully, even in this highly corrosive mixture, provided the film is perfectly intact. However, should a break occur, the coating will lose adhesion rapidly and will rapidly peel off. The possibility of discontinuities or breaks in the coating should be recognized when testing coatings for tank linings.

These are examples of a few pitfalls encountered in the selection and use of protective coatings. Unfortunately, there is no established way to eliminate these hazards. Most important are recognition of the variables encountered and use of tests to show, realistically, performance to be obtained under actual conditions.

Chemical resistance of a coating must be determined under actual conditions. Surface preparation and application either must be controlled or the exact conditions must be recognized so a coating can be selected to give optimum performance under surface preparation and application conditions of the job at hand.

Reference

1. Report on Surface Preparation of Steels for Organic and Other Protective Coatings. TP-6G—Surface Preparation for Organic Coatings. NACE Publication 53-1.

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Vinyl Plastisols—

(Continued From Page 128)

ing between 1 mil and 1/16-inch a so-called "continuity" tester is used. A description of this continuity tester is given in the report.

(1) Application, Technology, Physical Characteristics and Chemical Resistance of Polyvinyl Chlor-Acetates. Report of T-6 A. *Corrosion*, **10**, No. 10, 349-354 (1954) Oct.

Discussion by C. J. McBride, Houston:

Hot dip applications of plastisols do not present concrete evidence as to the bond being maintained while the coated article is in submerged service in corrosive environments. The bond can be merely a physical phenomenon and cohesive bond of the plastisol itself.

My question deals with the adhesive system. Are there at present any bonding agents that will satisfactorily maintain a bond between a plastisol and a large metal substrate while in submerged service at maximum recommended temperature? As a hypothetical example, could any plastisol adhesive manufactured at present maintain a strong bond between palstisol and metal during extended service in a pickle solution of muriatic acid at 160 F?

Reply by F. L. Scott and W. C. Hosford:

The ability of a plastisol/bonding primer system to retain its adhesion while in submerged service is a function of the service treatment of the metal prior to the application of the bonding primer, the composition of the primer, the composition of the plastisol, the film thickness of the plastisol applied and the nature of the solution in contact with the lining system. There are plastisol/primer systems that will retain their bond during extended service in many chemical solutions at temperatures up to 150 or 160 F. However, we do not know of any plastisol/primer system that will maintain a bond in muriatic acid at 160 F particularly at a concentration higher than 15 to 20 percent hydrogen chloride.

Questions by Mills Dean III, David Taylor Model Basin, Dept. of the Navy, Washington, D. C.:

1. In general terms what are the techniques for a cementing system in use today for bonding flexible or rigid vinyl sheeting in tank linings?

2. Does anyone to your knowledge have a solventless system in current use for bonding flexible or rigid vinyl sheeting?

Replies by F. L. Scott and W. C. Hosford:

The questions are somewhat outside of the scope of the paper, however, it may be desirable to include this discussion. Our comments are as follows:

1. When bonding vinyl sheeting to steel, the steel surface must be cleaned free of mill scale, rust, etc. Generally it is recommended that this be accomplished by grit or sandblasting. The steel surface is then immediately primed with the bonding cement and allowed to dry completely. The vinyl sheeting is cleaned with a ketone type solvent to remove mold parting compound and then a second coat of cement is applied to both the steel and the vinyl sheeting and allowed to dry until a tacky stage is reached. The sheeting is then warmed and pressed against primed steel surface. All seams are then covered with a narrow strip of vinyl sheeting.

2. To the best of our knowledge solventless cements are not in current use for bonding flexible or rigid vinyl sheeting.

Oak Ridge Industrial Isotope Course Offered

Applications are being accepted now for a two-week course in industrial isotope radiography at the Oak Ridge Institute of Nuclear Studies to be held at Oak Ridge, Tenn., May 5-16. It is designed to give supervisory and technical personnel facility in handling sealed isotope sources to use them safely and efficiently.

Brochures describing the course and application blanks are available from Ralph T. Overman, Chairman, Special Training Division, Oak Ridge Institute of Nuclear Studies, P. O. Box 117. Deadline for application is March 15.

CORROSION ABSTRACTS

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3. CHARACTERISTIC CORROSION PHENOMENA

3.5 Physical and Mechanical Effects

3.5.8, 3.7.4, 3.2.2, 2.3.7, 1.6

Stress Corrosion Cracking and Embrittlement. WILLIAM D. ROBERTSON, Editor. Book, December 4, 1956, 202 pp. John Wiley & Sons, Inc., 440 Fourth Ave., New York 16, N. Y.; Chapman and Hall Ltd., London.

Papers presented at a symposium arranged by Corrosion Division, The Electrochemical Society, Boston, October, 1954. Included are:

The Phenomena and Mechanism of Stress Corrosion Cracking, Julius J. Harwood; The Structure of Grain Boundaries, Bruce Chalmers; Structural Factors Associated with Stress Corrosion Cracking of Homogeneous Alloys, W. D. Robertson and R. Bakish; Stress Corrosion Cracking in Homogeneous Alloys, L. Graf; Metallurgical Aspects of Stress Corrosion Cracking in Aluminum Alloys, E. C. W. Perryman; The Mechanism of Stress Corrosion Cracking Observed in a Magnesium Alloy, D. K. Priest; The Role of Boundary Adsorption in Stress Corrosion Cracking, Rudolf Speiser and J. W. Spretnak; Stress Corrosion Cracking of Austenitic Stainless Steels in Aqueous Chloride Solutions, T. P. Hoar and J. G. Hines; The Phenomena of Stress Corrosion Cracking in Austenitic Stainless Steel, C. Edeleanu; Stress Corrosion Cracking of Mild Steels, R. N. Parkins; On the Mechanism of Chemical Cracking, U. R. Evans; Hydrogen in Iron and Its Alloys, W. R. Heller; Ductility of Steel with Varying Concentrations of Hydrogen, Taiji Toh and William M. Baldwin, Jr.; Laboratory Techniques for the Investigation of Stress Corrosion Cracking, H. R. Copson. 13379

3.5.8, 6.3.6, 2.3.7

Investigation in the Field of Stress-Corrosion of α-Brass in Ammonia Vapor. Pt. II. The Kinetics of Crack-Formation. (In German.) FRANZ AEBI. *Z. Metallkunde*, **47**, No. 6, 421-426 (1956) June.

Graphical representation of durability against initial stress in stress-corrosion tests is discussed and a mathematical relation developed.—BTR. 12740

3.5.8, 6.3.6, 4.3.3

The Stress Corrosion Cracking of Copper Alloys. Rept. II. On Straight Brass. M. SUGIYAMA, S. UEDA AND H. SAXOTA. *Corrosion Engineering*, **5**, No. 2, 81-85 (1956) April.

Investigation of stress corrosion cracking of straight brass containing zinc 20 to 50% and of influence of stress upon the electrode potential of 70/30 brass in aqueous ammonia.

It was demonstrated that alloys containing 35-40% zinc had lowest resistance to cracking in aqueous ammonia and alloys containing up to 26% zinc were susceptible to intergranular cracking, while brass with over 30% zinc suffered transcrystalline cracking.

There was the following relation between the rupture time (P) and the magnitude of stress (σ), $\sigma = CP^N$; the same relationship held between rupture time and concentration of ammonia.

Potential-time curves of stressed brass, compared with those of non-stressed specimen, have shown the less noble value immediately after the corrosive agent was added; then the potentials rapidly became more noble in aqueous ammonia.

The electrode potential of stressed brass, as expected, was less noble than

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AWWA—Journal, American Water Works Association, Amer. Water Works Assoc., Inc., 2 Park Ave., New York 16, New York.

BL—Current Technical Literature, Bell Telephone Laboratories, 463 West St., New York 14, New York.

BTR—Battelle Technical Review, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio.

CBEC—Centre Belge d'Etude de la Corrosion (CEBELCOR), 21 rue des Drapiers, Brussels, Belgium.

CDCT—Boletin, Centro de Documentacion Cientifica y Tecnica, Plaza de la Ciudadela 6, Mexico 1, D. F.

EL—Electroplating and Metal Finishing, 85 Udney Park Road, Teddington, Middlesex, England.

HB—Translations, Henry Brutcher, P. O. Box 157, Altadena, California.

IIM—Transactions of The Indian Institute of Metals, 31 Chowringhee Rd., Calcutta 16, India.

INCO—The International Nickel Co., Inc., 67 Wall Street, New York 5, New York.

JSPS—Japan Society for the Promotion of Science, Mr. Hayata Shigeno, Secretary, Committee of Preventing Corrosion, c/o Government Chemical Industrial Research Institute, 1 Chome Nakameguro, Meguro-Ku, Tokyo, Japan.

MA—Metallurgical Abstracts, Institute of Metals, 4 Grosvenor Gardens, London SW 1, England.

MI—Metallurgia Italiana, Associazone Italiana dei Metallurgia, Via Moscova 16, Milan, Italy.

MR—Metals Review, American Society for Metals, 7301 Euclid Ave., Cleveland 3, Ohio.

NALCO—National Aluminate Corp., 6216 West 66th Place, Chicago 38, Illinois.

NSA—Nuclear Science Abstracts, United States Atomic Energy Commission, Technical Information Division, Oak Ridge, Tennessee.

OTS—Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C.

PDA—Prevention of Deterioration Abstracts, National Research Council, 2101 Constitution Ave., Washington 25, D. C.

RPI—Review of Current Literature Relating to the Paint Colour, Varnish and Allied Industries, Research Assoc. of British Paint, Colour and Varnish Manufacturers, Paint Research Station, Waldegrave Rd., Teddington, Middlesex, England.

SE—Stahl und Eisen, Verlag Stahleisen, m.b.H., Breite Strasse 27 (Schliessfach 2590), Dusseldorf, Germany.

TIME—Transactions of The Institute of Marine Engineers, The Memorial Bldg., 76 Mark Lane, London E. C. 3, England.

ZDA—ZDA Abstracts, Zinc Developemnt Association, 34 Berkeley Square, London W. 1, England.

that of the non-stressed specimen in 3% sodium chloride solution.—JSPS. 12679

3.5.8, 6.3.15, 6.2.4

Fatigue Tests of Titanium Alloy and SAE 4340 Steel Specimens (Final Technical Report. Part 4: Report No. 34). W. L. STARKEY AND J. A. COLLINS. Ohio State Univ. Research Foundation, RF Project 518, Contract AF33(616)-209, Jan. 23, 1956, 55 pp.

The effects of fretting-fatigue damage on the fatigue endurance limit of Ti 140-A titanium and SAE 4340 steel are reported. Primarily two variables were investigated. One was fretting pressure and the second was number of fretting cycles. The tests were performed on rotating-beam fatigue testing machines equipped with Prot loading attachments. It was found that titanium is in general more sensitive to fretting than is steel. Titanium also exhibits more scatter in the test data than does the steel. A decrease in endurance limit is observed when either the fretting pressure or number of fretting cycles is increased. (auth).—NSA. 13391

3.5.8, 8.5.3

The Failure of a Welded Drying Drum by Caustic Embrittlement. G. SINES AND E. C. MCLEAN. *Mech. Eng.*, **78,** No. 12, 1105-1109 (1956) Dec.

Description of investigation of failure of a steel welded drying drum used in paper industry. Failure was caused by caustic embrittlement, decarburization during annealing and severe stress concentrations. Shell metal was an annealed low-carbon steel and head metal was a higher-carbon steel than the shell (with 0.07 nickel). Photomicrographs, diagrams. —INCO. 13170

3.7 Metallurgical Effects

3.7.3

The Effects of Moisture in the Coatings of Metal Arc Welding Electrodes. P. D. BLAKE. *Welder*, **25,** No. 126, 35-40 (1956) April-June.

Main sources of potential hydrogen present in arc atmosphere during welding are moisture in electrode coating, water of crystallization, structural OH groups in minerals comprising coating and atmospheric moisture. Amount of potential hydrogen present in an electrode coating may result in hydrogen embrittlement of weld metal laid down by the electrode. For certain electrodes some moisture in the arc zone is a necessary feature if best arc operating characteristics are to be maintained. Discussion of moisture content in electrodes includes reference to steel, nickel-molybdenum-vanadium low alloy ferritic and 0.8 nickel steel electrodes. Photos.—INCO. 12741

3.7.3

Behavior of Welded Built-Up Beams Under Repeated Loads. J. E. STALLMEYER, W. H. MUNSE AND B. J. GOODAL. Paper before Am. Welding Soc., Nat'l. Fall Mtg., Cleveland, October 8-12, 1956. *Welding J.*, **36,** No. 1, 27s-36s (1957) Jan.

Flexural fatigue tests were made on small all-welded beams fabricated from A373 steel and typical structural grade steel. All beams were fabricated by manual arc method using E7016 electrodes and back-stepping procedure. Typical butt-welded field splice configurations (either with or without cope holes) were investigated. Fatigue tests were also made on as-received mill specimens of A373 and on butt-welded joints, either parallel or perpendicular to stress direction. Presence of splice reduces fatigue strength of welded beam, the degree depending on splice configuration. Tables, diagrams.—INCO. 13472

3.7.3

Some Observations with the Electric Spark Machining Process. G. R. WILMS AND J. B. WADE. *Metallurgia*, **54,** No. 326, 263-268 (1956) Dec.

Investigation of spark machining of aluminum, antimony, chromium and iron showed that deformation of the metal surface is a basic feature of process and that cracks are produced in metals which are brittle at room temperature. These effects are removed in practical applications by polishing. Discussion of theory that metal removal is effected by thermal rather than purely mechanical means is given. Photomicrographs, 14 references.—INCO. 13431

3.7.3

How Heat and Time Affect Welding. A. C. WARD. Gen. Electric Co. *Iron Age*, **179,** No. 3, 75-77 (1957) Jan.

In each welding pass, temperatures at weld zone can jump to 2500 F and higher. This temperature changes steel and iron parts at the crystalline level, changing tensile strength and other mechanical properties. How these changes take place, when and where they occur and their magnitude depend on the carbon and alloy content of workpiece material, part thickness, heat input in welding, speed of travel, number of passes and rate at which workpiece cols. Isothermal Transformation Diagram plots time against temperature and by controlling cooling rate, moment when 1 crystalline structure changes into another can be predicted. Diagram can help pin down correct welding procedures and postweld heat treatment. ITD permits predicting time-at-temperature needed to bring about transformations and tells what to expect of metal adjacent to weld zone. It warns welder of formation of brittle martensite and calls attention to need for post-weld heat treatment to avoid failure in service through stress cracking. Photomicrographs, diagram.—INCO. 13181

3.8 Miscellaneous Principles

3.8.2, 3.5.9

Influence of Temperature on the Intensity of Diffusion Current and the Thickness of the Diffusion Layer. (In Russian.) V. V. GERASIMOV AND I. L. ROZENFEL'D. *Bull. Acad. Sci. URSS*, Classe Sci. Chim., No. 7, 779-783 (1956). 13202

3.8.2, 6.3.6, 4.3.2

Corrosion of Copper in Sulfuric Acid (Technical Report No. XVIII). NELDON L. JENSEN AND MILTON E. WADSWORTH. Utah Univ. Inst. for the Study of Rate Processes, Project NR-051-192, Contract N7-onr-45103, July 16, 1956, 60 pp.

The corrosion of copper in sulfuric acid solutions was investigated, and the rate of corrosion was measured by determining the amount of cupric ion in the solution at various periods of time and under known conditions. The rate was determined as a function of time, agitation of the solution, acid concentration, and partial pressure of oxygen. A mechanism was postulated that satisfied the experimental observations. The corrosion of copper was found to be linear over the range of time under all the conditions investigated and the rate was observed to be directly proportional to the partial pressure of oxygen. The rate, as a function of sulfuric acid, indicated an adsorption process in which the active adsorbate was undissociated sulfuric acid. Calculations of the energy of activation for the over-all process using the Arrhenius equation was found to be 14.2 koal per mole. The rate-determining portion of the process was considered to be the adsorption of undissociated sulfuric acid, followed by a rearrangement on the surface. The adsorbed species reacted with molecular oxygen and decomposed through an activated complex into the products of the reaction. The net reaction was considered to be $2\ Cu + H_2SO_4 + O_2 + 2H^+ \rightleftarrows 2\ Cu^{++} + SO^-{}_4 + 2H_2O$. The data were correlated by applying the Eyring theory of absolute reaction rates. (auth) — NSA. 13235

3.8.2, 6.3.11

Investigations on the Electrochemical Behavior of Gold. (In Italian.) G. POLI. *Gazz. Chim. Ital.*, **86,** 526-550 (1956) May-July. 13326

5. PREVENTIVE MEASURES

5.3 Metallic Coatings

5.3.3, 2.5

How Standards Can Serve as a Guide for Corrosion-Protection by Metallic Coatings. (In French.) P. MORISSET. *Corrosion et Anti-Corrosion*, **4,** No. 1, 22-35 (1956) Jan.

A paper presented to the study conference on metallic coatings organized in June, 1955 by the Association des Ingenieurs en Anti-corrosion de France et de l'Union Francaise. The French standards covering metal coatings, accelerated testing, fabrication, terminology and articles protected by coatings are listed and critically examined. Suggestions are made for a code of practice on the use of coatings and brief recommendations are given on the choice of protection in various conditions.—ZDA. 13288

5.3.4

Electroplating Chromium on Unusual Alloys. C. LEVY. Paper before Am. Electroplaters' Soc., 43rd Ann. Conv., Washington, D. C., June 17-22, 1956. *Tech. Proc. AES*, **1956,** 219-222.

Presents practical methods for electroplating chromium on molybdenum, Type 502 stainless, V-36, Inconel X, S-816, Hastelloy C, Hastelloy B, 19-9DL and Stellite 21. Photomicrographs of plated specimens are shown; heavy nickel deposit was applied to protect plate during polishing. Chromium plate on SAE 4340 is shown for comparison of adhesion. Thickness of chromium plate was 0.002-0.003 inch.—INCO. 13377

5.3.4

Conservation of Nickel in Electroplating. V. L. MCENALLY, JR. AND F. G. BRUNE. Naval Industrial Reserve Aircraft Plant. *Metal Progress*, **70,** No. 6, 89-92 (1956) Dec.

At least one-third of nickel used in electroplating is expended because of

poor distribution. Principle of current flow being greatest at parts of greatest curvature is considered. In addition to anode shape, distance between anode and cathode also affect variation in plate thickness. Methods for control of current distribution include use of "robber," a conductor near cathode in high-density area on which nickel is deposited; placement of shield between anode and cathode to force current to flow around it and decrease plate thickness at normal high point; use of shaped anode or inset anodes to counteract shape of cathode; and increasing anode-to-cathode distance.—INCO. 13287

5.4

Electrogalvanizing from the Pyrophosphate Bath. J. VAID AND T. L. RAMA CHAR. *J. Sci. Ind. Research* (India), Sec. B, **15,** No. 9, 509-515 (1956) Sept. Optimum conditions for electrodeposition of zinc from pyrophosphate bath established. Deposits comparable to those from cyanide bath; effect of various addition agents on quality of deposits and current efficiencies investigated.—BNF. 13403

5.9 Surface Treatment

5.9.4

Finishes for Aluminum Alloys. Pt. 1. Electrolytic or Anodic Coatings. Pt. 2. Chemical or Conversion Coatings. WALTER E. POCOCK. *Metal Progress,* **70,** Nos. 4, 5, 75-78, 97-101 (1956) Oct., Nov.

I. This is a review article detailing the processes used in applying anodic and chemical coatings on aluminum including pretreatment and post treatment such as sealing and dyeing.

II. This is a review article of the chemical or conversion coating finishes for aluminum alloys. These are mostly coatings formed from the chemical reaction of aluminum with chromic solutions. They provide excellent resistance to corrosion and good bases for paint. They are inexpensive to produce and have replaced anodic coatings in many applications. 13325

5.9.4

The Origin of the Hydrated Oxide Formed When Anodic Aluminium Oxide Films are Sealed in Boiling Water. I. S. KERR. *Proc. Phys. Soc.* (England), Sec. B, **69,** Pt. 10, No. 442B, 1055-1056 (1956) October 1.

Further new evidence on the origin of the Y-A100H, made possible by the ability of electron microscope to obtain diffraction patterns from particular areas of a specimen, are described. Aluminum foil (about 0.01 cm thick) was anodized at 50 V in an ammonium borate solution. Most of the aluminum was then stripped from the oxide with chlorine at 300 C, leaving a metal foil network sufficiently rigid to support the unaffected oxide film across windows from which the metal was completely removed. As the oxide film thickness was about 700 A, it was possible to penetrate it with an electron beam without unduly large incoherent scattering. Before sealing the film was examined in the electron microscope. When adjusted for diffraction the expected halo pattern was obtained. The foil was then sealed by immersion in boiling distilled water for 20 minutes and returned to the microscope. Certain areas gave a halo pattern identical with the original, while other areas gave a sharper boehmite pattern. Closer examination proved that the halo patterns originated from the center of large areas of oxide film or from tongues which projected from the metal but had otherwise free edges. The boehmite pattern came from parts of the oxide film near the edges of the metal or from regions where small specks of aluminum were dispersed over the film. It is thus concluded that the boehmite is formed only in the immediate neighborhood of aluminum.—ALL. 13249

5.9.4

Notes on Post-Treatment for HAE Coatings. H. SALMON AND F. OGBURN. *Plating,* **43,** No. 10, 1241-1243 (1956) October.

Of seven post treatments which have been tested, two are recommended: immersion in ammonium fluoride · hydrofluoric acid/sodium dichromate solution, air drying and immersion in sodium chromate solution; or treatment in ammonium di-hydrogen orthophosphate/sodium chromate solution.—BNF. 13350

5.9.4

Water Sealing of Detached Aluminium Oxide Anodic Film. ROY C. SPOONER. *Nature,* **178,** No. 4542, 1113-1114 (1956) Nov. 17.

Aluminum sheet (99.87%) of 0.5 mm thickness was anodized (15% weight sulfuric acid, 21 C, 1.6 amp./sq. dm., 60 min.) to form an oxide film approximately 0.030 mm thick. The weight gain on water sealing (deionized water 100 C, pH 6.1, 30 minutes) of such coatings was determined. The film weight was determined by dissolution of the coating in a chromic-phosphoric acid solution. Panels anodized in a similar manner were sheared into 1 cm by 2 cm squares and the unsealed oxide film was detached from these by a modified Wernick technique (5-10 grams mercuric chloride/100 mm ethyl alcohol). The detached films were rinsed twice in absolute methyl alcohol. The weight gain on water sealing of detached oxide film was determined and compared with that of the non-detached film. Electron diffraction patterns were obtained on non-detached and detached oxide films, in both the unsealed and water-sealed conditions. The weight gain on sealing the film, which was sealed after being detached (7.1%), was in good agreement with the value obtained for the non-detached. The similarity of the results obtained with films detached from their aluminum base to those for non-detached films confirms that water sealing of sulfuric acid type anodic films is a hydration process and does not require the presence of metallic aluminum.—ALL. 13381

6. MATERIALS OF CONSTRUCTION

6.2 Ferrous Metals and Alloys

6.2.2, 3.5.8

The Influence of Understressing on the Fatigue Properties of Flake Graphite and Nodular Graphite Cast Irons: BCIRA Res. Aept. No. 447. G. N. J. GILBERT AND K. B. PALMER. *J. Res. and Development, Brit. Cast Iron Res. Assoc.,* **6,** No. 9, 410-421 (1956) Dec.

Understressing for 20 million cycles at a stress of 0.5 ton/sq. in. below fatigue limit, has not significantly changed fatigue limit or fatigue life at stresses

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above the fatigue limit, of series of pearlitic and ferritic flake graphite and nodular graphite irons. Flake graphite irons tested were unalloyed and in pearlitic condition a high tensile strength of 20.9 ton/sq. in. was obtained. Nodular irons tested were typical of good as-cast pearlitic and heat-treated ferritic irons; they contained 0.66 and 0.67 nickel. Other investigators reported increases in fatigue limit in excess of 30% due to understressing, and further tests on lower strength irons tested by these investigators are proposed, to determine whether such increases in fatigue limit are confirmed. Tables summarize chemical composition, tensile properties, and fatigue results for notched and un-notched specimens. Photomicrographs, graphs.—INCO. 13243

6.2.2, 8.3.1

Wear Tests of Plowshare Materials. N. MOHSENIN, H. L. WOMOCHEL, D. J. HARVEY AND W. M. CARLETON. *Michigan State Univ. Agric. Eng.*, **37**, No. 12, 816-820 (1956) Dec.

Fact that annual consumption of plowshares is in the millions and that plowshare maintenance constitutes 10-20% of plowing cost, prompted investigation of role of alloying elements (especially carbon) as factor in wear resistance and suitability of nodular iron as plowshare material. Experimental samples were made from 11 types of irons to include ordinary cast iron and nodular iron of high or low carbon and alloyed and unalloyed compositions (chromium, nickel and molybdenum alloying constituents) made in chilled and unchilled conditions. Correlation between accelerated laboratory wear test and field test results and correlation between hardness and weight loss in field are discussed.—INCO. 13261

6.2.3, 3.5.8

The Fretting Corrosion of Mild Steel. J. S. HALLIDAY AND W. HIRST. *Proc. Royal Soc.*, Ser. A, **236**, 411-425 (1956) August 2.

Measurements of the frictional forces, the degree of damage and the variations in the electrical contact resistance for a wide range of applied loads, vibration amplitudes and number of cycles of motion.—MR. 13236

6.2.3, 8.1.2

New Aspects of Rust Protection in Modern Building. R. MARPON. *Peintures, Pigments, Vernis*, **32**, No. 7, 604-608 (1956).

The protection of steel sheet for prefabricated buildings is chiefly considered. Surface preparation and choice and execution of painting schemes are covered. —RPI. 13262

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.9

Arc-Cast Molybdenum Base Alloys. M. SEMCHYSHEN AND R. Q. BARR. *Aviation Age*, **26**, No. 5, 34-41 (1956) Nov.

Molybdenum alloys are getting more attention as possible materials to meet increasingly stringent requirements of high performance aircraft and missiles. At present, molybdenum is the only base material for the 1800-2200 F range because it has a melting point of 4750 F, as against 3000 F for other high temperature alloys. Creep-rupture tests showed that at 1600 F molybdenum alloys are markedly superior over high-temperature alloys such as S-816. Tests on relationship between alloy content and rupture strength showed that this was non-linear for molybdenum-titanium materials and that a 0.5 titanium content provided highest strength. Major problem to be overcome before molybdenum can be used in high temperature applications is its very high oxidation rate at these temperatures. Chromium, nickel and silicon coatings on molybdenum prepared under hydrogen showed poor oxidation resistance while those prepared under argon had good resistance. Tables showing creep-rupture strength of wrought, arc-cast molybdenum and molybdenum base alloys in stress-relieved and fully recrystallized condition includes nickel alloys. Graphs.—INCO. 13361

6.3.10

Development and Testing of Vacuum Melted Nickel—Molybdenum Alloys with Minor Alloying Additions (Final Report). O. PRESTON, N. J. GRANT AND C. F. FLOE. New England Materials Lab., Inc. for Oak Ridge Nat'l. Lab. U. S. Atomic Energy Comm. Pubn., ORNL-2181, October 18, 1956, 40 pp. Available from: Office of Technical Services, Washington, D. C.

A series of five simple vacuum melted nickel-molybdenum alloys in which the molybdenum increased progressively from 17 to 36% is discussed. Air melted Hastelloy B was included for comparison purposes. The test program included (for 3 heat treatments) creep rupture tests up to about 500 hours at 1350, 1500 and 1650 F, room temperature tensile tests, and room temperature tensile tests after 100 hours exposure at 1500 F to determine the degree of embrittlement. Because of severe embrittlement at room temperature after high temperature exposure of these simple binary nickel-molybdenum alloys, two of the intermediate molybdenum compositions were selected and additions of chromium, iron, vanadium and niobium were made to determine the degree of stabilization conferred by these various addition elements. (auth).—NSA. 13323

6.3.10, 2.2.2, 6.2.5

Atmospheric Corrosion Behavior of Some Nickel Alloys. H. R. COPSON. Paper before Am. Soc. Testing Materials, 58th Ann. Mtg., Symp. on Atmospheric Corrosion of Non-Ferrous Metals, Atlantic City, June 29, 1955. ASTM Special Technical Pubn. No. 175, 1956, 141-158.

Weight loss and tension data are presented for nickel and Monel specimens exposed to various marine, industrial and rural atmospheres in ASTM 20-year test program. Data on atmospheric exposure tests carried out independently at Bayonne on nickel, Monel, some nickel alloys and stainless steels are presented. These show the effects of alloying nickel with copper, iron and chromium and effects of shelter on other variables. In nickel, nickel-copper alloys and nickel-iron alloy high in nickel, attack is uniform and corrosion rate constant with time. High chromium alloys show local pitting attack. Sheltered specimens corrode more than outright exposed ones and screen and wire corrode faster than sheet. Behavior of nickel-copper alloys varies with location. In nickel-iron series up to 20% iron additions decreased corrosion in industrial location; with more iron corrosion increased. Heat treatment and surface condition are of minor importance compared to chromium content as factors determining behavior of stainless steel. Numerous table and graphs.—INCO. 12769

6.3.10, 8.8.1

New Chemical Products and High Nickel Alloys. H. O. TEEPLE AND R. M. FULLER. *Ind. & Eng. Chem.*, **48**, No. 11, 1971-1974 (1956) Nov.

Discussion of application of nickel and high nickel alloys in chemical and process industries for economical corrosion resistance and for protection against product contamination includes Duranickel, Monels, Inconels, Hastelloy and Chlorimet alloys. Corrosion, mechanical and physical properties of high nickel alloys are discussed in their application to chlorine, iodine, fluorine, bromide, alkalies, pharmaceuticals, synthetic detergents and acid processing. Tables, photos, 30 references.—INCO. 13397

6.3.11, 3.2.2

The Effect of Internal Oxidation on the Tensile Properties of Some Silver Alloys at Room and Elevated Temperatures. E. GREGORY AND G. C. SMITH. Sintercast Corp. *J. Inst. Metals*, **85**, Pt. 3, 81-87 (1956) Nov.

Conditions affecting production of nonmetallic phases by internal oxidation of silver alloys containing silicon or aluminum were investigated. A very marked inhibition of grain growth and recrystallization by the disperse phase was obtained. Short-time tensile tests at 18, 200, 340, 410 and 590 C on 0.13 silver-silicon, 0.05 silver-aluminum and 0.25 silver-aluminum alloys showed a considerable improvement in strength after internal oxidation. Strength was dependent on degree of dispersion of the oxide, the greatest improvement being noted in specimens containing finest dispersion. Graphs, 13 references. —INCO. 13221

6.3.11, 3.2.3

The Scaling of Technical Gold Alloys and Its Avoidance in Heat Treatment. (In German.) H. SPENGLER. *Metall*, **10**, No. 13/14, 617-620 (1956) July.

Deals with scaling processes in 12- to 14-carat gold-silver-copper alloys with and without additions of palladium, nickel and zinc. Silver and palladium decrease the rate of scaling in alloys, while nickel and zinc increase it.—BTR. 12602

6.4 Non-Ferrous Metals and Alloys—Light

6.4.2, 3.5.8, 2.3.7

The Effect of Stress Concentrations on the Fatigue Resistance of a Duralumin Type Aluminium Alloy. J. Y. MANN. *J. Roy. Aeronaut. Soc.*, **60**, No. 550, 681-686 (1956) Oct.

Results of tensile and fatigue tests carried out at Aeronautical Research Laboratories, Melbourne, on notched and unnotched specimens of solution heat treated, aged and cold rolled aluminum-4.4 copper-0.6 manganese-1.5 magnesium-0.1 chromium to find a basis on which to assess notch sensitivity. Conclusions show strength reduction factor for a particular notch depends on endurance at which it is measured; greatest reduction in fatigue strength is not caused by sharpest notch.—BNF. 13289

6.4.2, 3.5.8, 2.3.7, 3.7.2

Stress-Corrosion of Wrought Ternary and Complex Alloys of the Aluminum-Zinc-Magnesium System. R. CHADWICK,

N. B. MUIR AND H. B. GRAINGER. *J. Inst. Metals*, **85**, Pt. 5, 161-170 (1957) Jan.

Previous investigations of stress-corrosion in aluminum-zinc-magnesium alloys are briefly reviewed. Object of present investigation was to develop a method of test under closely defined conditions and to use this to establish stress/time-to-failure relationships for number of aluminum-zinc-magnesium alloys. New equipment designed for direct-tension stressing, using spring loading combined with dash-pot device to absorb energy released by breaking of stressed specimen, permits installation in small space of several units working at high loading. In alloys with 5-9 zinc and 0.4-2.8 magnesium, stress-cracking occurs at comparatively low stresses, time-to-failure diminishing with increase in zinc plus magnesium content. Complex alloys with 7 zinc and 2.25 magnesium with smaller additions of copper, manganese and chromium, were tested. Increased resistance to stress-corrosion conferred by iron and silicon in commercial purity metal is ascribed to grain refinement. Copper is beneficial in that it develops anodic areas at grain centers; chromium diminishes stress-corrosion tendencies by producing elongated grain structure. Tables, graphs, photomicrographs.—INCO.
13461

6.4.2, 3.5.8, 3.7.4

Stress-Corrosion of an Aluminum Alloy. A. J. MARTIN. *Metal Industry*, **89**, Nos. 25, 26, 511-515; 531-532, 540 (1956) Dec. 21, Dec. 28.

Susceptibility to mechanical embrittlement at grain boundaries and to localized, grain boundary corrosion attack and a highly negative electrode potential leading to a high, general corrodibility and to presence of local cells of large potential difference lead to overall susceptibility to stress corrosion of aluminum-zinc-magnesium alloy. Microscopical examination of corroded specimens indicated that such cells were present at grain boundaries and that acid, liberated in these regions, accentuated susceptibility to boundary corrosion. Diffusion of zinc, magnesium and copper to grain boundaries was primary cause of severe intergranular corrosion and brittleness found after heat treatments. Further ageing led to an increased resistance to intergranular fracture and a correspondingly increased resistance to stress corrosion, even though corrosion attack was still concentrated at grain boundaries. Photomicrographs, graphs, 39 references.—INCO.
13285

6.4.2, 3.7.2

Influence of Small Amounts of Alloying Elements on the Technological Properties of Aluminium. (In Hungarian.) D. ANDRAS. Kohaszati Lapok (*Hungarian J. Metallurgy*), **11**, No. 4, 185-187 (1956) April.

Surveys the effect of 25 elements on electrical conductivity, corrosion resistance, grain size and mechanical properties of aluminum. 22 references.—BNF.
12760

6.4.2, 3.7.2, 7.3

Influence of Iron and Silicon Contents on the Corrosion Resistance of A-U2GNZr. (In French.) ROGER CHEVALIER AND ROGER LERNER. *Rev. Aluminium*, 33, No. 233, 609-613 (1956) June.

Results show that the most notable effects of corrosion on turbo-jet compressor blades is a reduction of the elongation; anodization offers a very effective protection but tension fosters corrosion.—MR.
12766

6.4.2, 3.7.3

Effect of Quenching and Ageing in the Aluminium-Magnesium Alloys Containing Silicon as Either an Impurity or a Secondary Constituent to the Extent of 0-1%. J. RIGAL AND M. RENOUARD. *Rev. Met.*, **53**, No. 4, 263-269; disc., 269-270 (1956).

It is known that the mechanical properties of aluminum containing $>6\%$ magnesium are greatly improved by quenching followed eventually by ageing, even in the absence of impurities or secondary elements; the magnesium goes completely into solution at temperatures below 450 C. With alloys containing $<6\%$ magnesium, no appreciable hardening by precipitation is observed unless the alloy is heated to temperatures considerably >500 C. before quenching, although theoretically all the magnesium should go into solution just above 300 C. Experiments were carried out on a high purity aluminum-magnesium alloy (A_9-G_3) and three alloys made by adding 0.2% silicon, 0.2% iron, or 0.2% silicon + 0.2% iron, to this. The alloys were quenched from between 475 and 575 C and aged at 170-180 C for various times. Only in the presence of silicon was there any notable improvement in the mechanical properties, the best period for agenig being ~20 hr. There appears to be a close relation between the hardening and the amount of silicon that goes into solution, which occurs at 530-580 C. Provided that this ageing treatment does not lower the corrosion-resistance (which it probably would for the 4-5% magnesium alloy), it should prove an advantage for the 2-3% magnesium alloy containing a little silicon.—MA.
13341

6.4.2, 3.7.3

Studies on Corrosion of Welded Aluminum and Its Alloy Plates. H. YAMAGUCHI AND J. YAMAMOTO. *Light Metals* (Japan), No. 19, 76-79 (1956) May.

This report deals with the studies on stress corrosion of argon welded ANP (aluminum-4% magnesium-0.5% manganese-0.2% chromium) plates dipped in brine and aqueous solution of 3% sodium chloride + 0.1% hydrogen peroxide.

Stress corrosion cracking did not occur in any case. The electrode potentials of deposited zone measured in the aqueous solution of 3% sodium chloride + 0.1% hydrogen peroxide are more noble than those of mother plates.

Corrosion occurs especially on the heat affected zone of the mother plates, but very little on the deposited zone.

Corrosion of the heat affected zone of the plates in the case of oxyacetylene gas welding is more remarkable than that in the case of argon welding.—JSPS.
12655

6.4.2, 3.7.3, 3.5.8

The Effect of Heat Treatment on the Susceptibility of Sand Cast Aluminum Alloy 220 to Stress Corrosion Cracking: Topic of the Month. FRED M. REINHART. *Corrosion*, **13**, No. 1, 1t-2t (1957) Jan.

Examination of 10% magnesium-aluminum sand casting that failed after short service life in marine atmosphere, showed intergranular and branching cracks, typical of stress-corrosion cracks in aluminum alloys. As-received and heat treated specimens were stressed in tension and immersed in NaCl-H_2O_2 solution. Results show that susceptibility to stress corrosion cracking is decreased appreciably when rate of quenching from solution heat treating temperature is increased. Photomicrograph.—INCO.
13192

6.4.2, 4.3.6, 4.4.10, 3.6.2

The Corrosion of 2S Aluminum in Sodium Borate and Lead Acetate Solutions. JAMES L. ENGLISH. Oak Ridge National Lab. U. S. Atomic Energy Comm. Pubn., ORNL-173, October 14, 1948 (Declassified Jan. 5, 1956), 6 pp. Available from: Office of Technical Services, Washington, D. C.

Static corrosion tests were conducted with annealed 2S aluminum panels exposed to 5 percent borax solutions and 25 percent lead acetate solutions at room temperature. Corrosion attack was severe in the lead acetate solutions (1.7 mils/month) and less severe in sodium borate solutions (0.31 mils/month). In all cases, corrosion damage appeared to be the result of direct chemical attack and was generally of a uniform nature with very minor localized attack. Waterline attack was studied by partial immersion of the specimens in the corroding media and was of minor consequence in the borax solutions but was very pronounced on the panels immersed in lead acetate. Various organic-type protective coatings were employed to prevent corrosion attack on 2S aluminum by lead acetate solutions. Bakelite lacquer and Pruf-coate were found to be the most resistant. (auth).—NSA.
12727

6.4.2, 5.2.1

Electrochemical Protection of Duralumin. (In Russian.) V. V. GERASIMOV AND I. L. ROZENFEL'D. *J. Phys. Chem., USSR* (Zhur. Fiz. Khim.), **30**, No. 8, 1816-1820 (1956) August.

Effect of protective circuit potential on the speed and depth of corrosion penetration.—MR.
13218

6.4.3

Beryllium Offers High Structural Promise. G. A. HOFFMAN AND W. R. MICKS. Paper before Inst. Aeronautical Sciences, San Diego, August 15-17, 1956. *Aviation Week*, **65**, No. 25, 57, 59-61 (1956) Dec. 17.

Study of characteristics of beryllium indicates that it offers high promise as an aircraft and missile structural material. Advantages seen for transport plane are possible airframe weight reduction of about 50% in comparison with aluminum alloy structures, range increase of about 40% and metals 'worth-in-use' to range as high as $1000 pound. Properties of beryllium are compared to aluminum, 17-7PH stainless and titanium alloy. Problems encountered in use of beryllium are discussed. Graphs.—INCO.
13239

6.4.3

Beryllium Thermal Stress Test. H. H. WINKLER. Knolls Atomic Power Lab. U. S. Atomic Energy Comm. Pubn., KAPL-M-HHW-2, September 19, 1949 (Declassified February 9, 1956), 10 pp. Available from Office of Technical Services, Washington, D. C.

Thermal stress tests were made on beryllium wafers in liquid sodium for a period of 140 hours with Δt ~300 F. A sampling of the wafers were metallographed, indicating beryllium cracks under high thermal stress.—NSA.
13364

6.4.3

Thermal Stress in a Beryllium Washer. WARREN F. WITZIG AND WILLIAM A. RIEMEN. Knolls Atomic Power Lab. U. S. Atomic Energy Comm. Pubn., KAPL-

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M-WW-2, July 1, 1949 (Declassified February 8, 1956), 17 pp. Available from Office of Technical Services, Washington, D. C.

Thermal stress was produced by a large radial heat flux in a beryllium washer immersed in a NaK alloy. After 7000 cycles of thermal stress, the washer maintained its principal dimensions but developed small radial cracks. There was a 2 mg wt gain despite obvious corrosion.—NSA. 13444

6.4.3, 3.7.3

Corrosion Resistance of Hot-Pressed and Extruded Beryllium. JAMES REED. Oak Ridge Nat'l. Lab. U. S. Atomic Energy Comm. Pubn., CF-50-5-81, May 21, 1950 (Declassified February 20, 1956), 15 pp.

Corrosion tests of 60 days' duration were run to determine the relative corrosion resistance of hot-pressed and extruded beryllium exposed to distilled water at 850 C. The solution was 0.005 M in hydrogen peroxide and a pH range of 5.5 to 6.5 was maintained. All the beryllium samples were machined and/or ground in different ways so that the effect of cold working on the corrosion resistance could be studied. (auth).—NSA. 13317

6.4.4

Tensile Properties of HK31XA-H24 Magnesium-Alloy Sheet Under Rapid-Heating Conditions and Constant Elevated Temperatures. T. W. GIBBS. U. S. National Advisory Cttee. for Aeronautics, Tech. Note 3742, August, 1956, 20 pp.

Specimens of magnesium-alloy sheet (magnesium-3 thorium-0.72 zirconium) rolled and partially annealed, were heated to failure at nominal temperature rates from 0.11 to 55.5 C/sec. under constant load conditions. Rapid-heating yield and rupture stresses are compared with yield stress-strain tests for half-hour exposure. Linear temperature-rate parameters used.

8. INDUSTRIES

8.2 Group 2

8.2.1, 7.7

Corrosion: The Constant Enemy of Metals. A. MENDIZZA, *Bell Labs Record*, **34**, No. 12, 451-455 (1956) December.

Discussion of corrosion encountered in telephone equipment from atmospheric moisture and chemicals in different locations. Comparison of field and laboratory tests of galvanic corrosion of Types 430 and 302 stainless, nickel and copper is given. Corrosion of underground and submarine cables is discussed. Photos.—INCO. 13282

8.2.2, 4.5.1, 3.6.9

Corrosion Control on Underground Structures in Power Plants. R. M. WAINWRIGHT AND D. P. PRICE. Paper before Am. Power Conf., 18th Ann. Mtg., Chicago, March 21-23, 1956. *Proc. Am. Power Conf.*, **18**, 474-481 (1956).

Corrosion of metal structures buried in soil around and under power plant proper and in plant and substation yards is caused by soil corrosion due to single metal and multimetal galvanic cells and by stray current corrosion. Corrosion control by coatings and cathodic protection is discussed. Corrosion control on an older power plant and a new power plant is described—Diagrams.—INCO.

8.2.2, 4.6.2

Avon No. 8: A Supercritical Pressure Plant. C. A. DAUBER. Paper before Am. Soc. Mech. Engrs., Semi-Annual Mtg., Cleveland, June 17-21, 1956. *Combustion*, **28**, No. 2, 47-56 (1956) August.

Description of 250,000-kw supercritical pressure plant. Control of pH by addition of ammonia or hydrazine at the hotwell condensate pump and deaeration of condensate protect metals in the preboiler cycle. Filters remove corrosion products in the condensate. Tests on Types 316 and 347 stainless showed that hot ductility of fine grain Type 316 is better for use for piping for the eight main steam boiler leads. Walls of station are constructed of insulated stainless steel panels. Diagrams.—INCO. 12755

8.3 Group 3

8.3.4

Non-Ferrous Metals in the Brewery. (In German.) R. RUSSE. *Metall*, **10**, No. 17/18, 818-821 (1956) September.

Use of non-ferrous metals discussed in relation to the various departments of a brewery: malting, brewing, cooling, fermentation, storage, cleaning and filling of casks, internal and external transport.—BNF. 13354

8.4.2

Deterrence of Hydrogen Blistering. B. W. NEUMAIER AND C. H. SCHILLMOLLER. *Corrosion Technology*, **3**, 357-361 (1956) Nov.

Major protective steps taken at fluid-unit gas plant were early removal of hydrogen peroxides and extensive water-washing, starting at the fractionator overhead system.—BTR. 13.04

8.4 Group 4

8.4.3

Choosing Metals for the Refinery. J. J. B. RUTHERFORD. Babcock & Wilcox Co. *Petroleum Processing*, **11**, No. 12, 52-56 (1956) December.

Major component of all processing equipment in oil and petrochemical field is steel. Carbon steel is the cheapest and most versatile of the wrought products, nickel steels are used for extra low temperature service, stainless for still lower temperatures and chromium-molybdenum alloys for service between ordinary and stainless steels. Mechanical strength of steels and applications to design equipment are discussed. Table gives comparison of allowable stress values for Types 304, 321, 316 and 310 stainless steels at high temperatures. Photomicrographs show failures of stainless, Croloy 5 and nickel-chromium alloy and steel by oxidation, sulfur compounds and hydrogen. Penetration of zinc into grain boundaries of stainless steel at high thermal stress shows why steel should not be fabricated for elevated temperature by copper, brass or bronze tools.—INCO. 13383

8.4.3

Corrosion Prevention in Gas Condensate and Sweet Oil Wells. M. BOCK. Sun Oil Company. *Petroleum Engr.*, **29**, No. 1, B111-122 (1957) Jan.

Discussion of causes and detection of corrosion in tubing, casing and other well equipment and fundamentals of determining extent of corrosion and economic use of materials, methods and inhibitors to prevent damage. Corrosion in oil wells depends on water to oil ratio, rate and method of production and amount of carbon dioxide and organic acids in water. Gas-condensate wells corrode because they produce acid water caused by carbon dioxide and organic acids. Prevention of corrosion by use of chemical inhibitors, protective coatings, by selection of proper equipment, by practicing proper operating techniques and by use of corrosion resistant alloy materials is discussed. Corrosion resistant materials given include 5 and 9 nickel and 9 and 12 chromium alloys. Photos, graphs.—INCO. 13193

8.4.3, 5.4.5

How to Paint a Drilling Rig. J. W. NEE, *Briner Paint Mfg. Co. World Oil*, **144**, No. 1, 121, 123-124 (1957) Jan.

Outlines types of coatings, their properties, applications and service to be expected. Included are oleoresins, alkyd resins, vinyl resins, chlorinated rubber, epoxy resins and butadiene-styrene copolymers.—INCO. 13440

8.6 Group 6

8.6.3

Corrosion in the Textile Industry. R. I. MAMUZIC. *Zastita Materijala*, **4**, No. 5, 165-167 (1956).

A general discussion, in which brief reference is made to the use of anti-corrosive paints.—RPI. 13264

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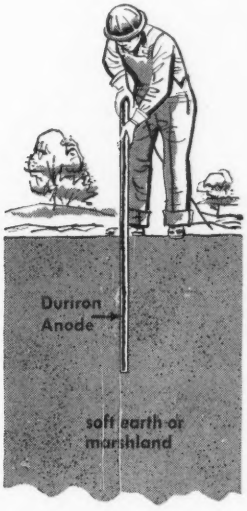

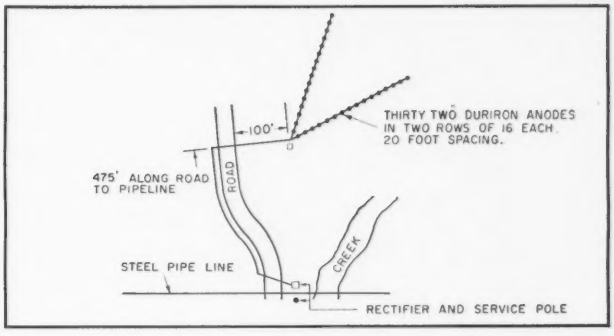
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